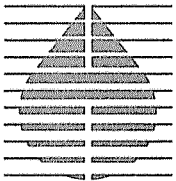


May 20, 1988

PART 2  
REMEDIAL INVESTIGATIVE WORK  
QUALITY ASSURANCE PROJECT PLAN  
MONTROSE SITE  
TORRANCE, CALIFORNIA



HARGIS+ASSOCIATES, INC.  
Consultants in Hydrogeology

Tucson/Phoenix/San Diego



HARGIS + ASSOCIATES, INC.

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May 20, 1988

Prepared by  
Hargis + Associates, Inc.  
for  
Montrose Chemical Corporation

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**PART 2**  
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**QUALITY ASSURANCE PROJECT PLAN**  
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**PART 2**  
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**1.0 INTRODUCTION/OVERVIEW**

This Quality Assurance Project Plan (QAPP) has been prepared in accordance with the U.S. Environmental Protection Agency (EPA) guidance document "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80", December 29, 1980, for the Part 2, Remedial Investigative Work (RIW) at the Montrose Chemical Corporation site (the Montrose site), in Torrance, California. In an ongoing effort to improve the quality of project work, this QAPP will be adhered to for all Part 2 RIW tasks involving future field work and for all Part 2 data reduction activities in support of the remedial investigative report, unless superseded by a future QAPP. Adherence to the quality assurance/quality control (QA/QC) program outlined in this document will ensure that the data collected are precise, accurate, complete, and representative. Quality assurance (QA) is defined as the integrated program designed for ensuring reliability of monitoring and measurement data. Quality control (QC) is defined as the routine application of standard procedures to obtain prescribed standards of performance in the monitoring and measuring process.

Quality assurance procedures such as tracking, reviewing, and auditing must be implemented to ensure that field and laboratory data are of high quality and that all project work is performed in accordance with professional standards, EPA and other governmental regulations and guidelines, and specific project goals and requirements. Outlined in this QAPP are the procedures to be followed to ensure the quality and reliability of data collected in the study area.



The QAPP covers each of the following activities:

- .. Monitor well installation.
- .. Groundwater, soil and sediment sampling; sample handling, packaging, and shipping; sampling safety and decontamination procedures.
- .. Field measurements including those related to lithologic logging, water level and water quality field measurements and the sediment thickness survey.
- .. Sample control/chain-of-custody.
- .. Field equipment calibration, operation and maintenance.
- .. Analytical procedures.
- .. Disposal of investigation-derived material.



## 2.0 PROJECT DESCRIPTION

The Montrose site occupies about 13 acres in Torrance, California (Figure 1). The area is bounded by the Southern Pacific railroad (SPRR) right-of-way and Normandie Avenue on the east, Jones Chemical Company and Los Angeles Department of Water and Power (LADWP) property on the south, a vacant lot on the west, and the McDonnell-Douglas facility on the north. The surrounding area consists of mixed residential, commercial, and industrial zones.

Between 1947 and 1982, the Montrose Chemical Company (Montrose) operated a dichloro-diphenyl-trichloroethane (DDT) manufacturing facility at the Montrose site. Although the use of DDT was banned in the United States in 1972, its use was not banned in other countries. Montrose continued to manufacture and export DDT until 1982, when the facility was closed and subsequently dismantled. The Montrose site is now capped with asphalt.

Initial investigations addressing the potential for contamination at the Montrose site included on-site sampling of soil and groundwater as well as off-site sampling of soil, sediment, and surface water. These investigations were conducted prior to May 1985 by the EPA and its contractors, the California Department of Health Services (DOHS), the Regional Water Quality Control Board, and by Hargis + Associates, Inc., acting on behalf of Montrose.

In June 1985, EPA contractor Metcalf & Eddy, Inc. conducted a preliminary on-site investigation referred to as Part 1 of the Remedial Investigation, which included soil sample collection from 17 soil borings and groundwater sample collection from five monitor wells constructed by Hargis + Associates, Inc. in April 1985. In March 1986, Metcalf & Eddy, Inc. released a Draft Preliminary Report summarizing their findings from the June 1985 study (Metcalf & Eddy, Inc., 1986). The report included recommendations on additional field data requirements as well as the current list of Target Chemicals.



In October 1985, a Consent Order between the EPA and Montrose concerning the performance of additional investigative activities was finalized. The off-site and on-site tasks were based on Metcalf & Eddy, Inc. (1984), as modified by Appendix A of the Consent Order. The tasks were designed to obtain information necessary for the performance of a Feasibility Study. The Part 2, Phase 1 tasks were performed during the period April 1986 to January 1987 and included off-site soil, sediment, and surface water sampling, on-site soil sampling, construction of monitor wells, and groundwater monitoring of the Bellflower aquitard and the Gage aquifer. Samples were analyzed for contaminants designated by EPA as Target Chemicals (Table 1).

Unless superseded by a future QAPP, this QAPP will be applicable to all Part 2 RIW tasks. Additionally, this QAPP specifically addresses the off-site sediment and off-site and on-site soil and groundwater tasks referred to as the Part 2, Phase 2A Remedial Investigation field activities (Phase 2A). Phase 2A will be performed for Montrose under the direction and supervision of Hargis + Associates, Inc., subject to review and approval by the EPA.

The objective of Phase 2A is to further determine the concentrations and areal extent of Target Chemicals in the groundwater, soil, and Dominguez Channel sediments which may have been caused by activities at the Montrose site, and to gather sufficient data of adequate quality to support the Feasibility Study. A detailed description of the proposed Phase 2A field program is provided in the accompanying Phase 2A Groundwater, Soil, and Sediment Sampling Plan (SAP) (Hargis + Associates, Inc., 1988).



### 3.0 PROJECT ORGANIZATION AND RESPONSIBILITY

The individuals responsible for each element of the overall program are identified in the Quality Assurance Organizational Chart for the Sampling Plan (**Figure 2**). The key individual responsible for quality assurance is the QA Project Manager. The QA Project Manager for this project is Mr. David Mohrbacher of Hargis + Associates, Inc. Meetings between the Project Director, the QA Manager, and the Project/Task Manager will be held as needed to review QA activities. These individuals are responsible for ensuring the collection of valid measurement data and for assessing measurement systems on a routine basis for precision and accuracy.



#### 4.0 QUALITY ASSURANCE OBJECTIVES

The overall QA objectives are to develop and implement procedures for obtaining and evaluating data that can be used to assess site hazards and develop alternative remedial actions, and that is defensible in a court of law. In order to provide defensible data, it is necessary that all measurement data have an appropriate degree of accuracy and reproducibility. All samples collected and all field measurements made must be representative of actual field conditions. Specific quality assurance objectives have been established for accuracy, precision, and completeness of analytic results. Accuracy shall be evaluated in spiked samples in terms of percent recovery. Precision shall be evaluated for duplicate samples in terms of percent difference. Completeness of analytic results will be evaluated by comparing the number of defective results to the actual number of analyses requested. Accuracy, precision, and completeness are defined in **Section 14, Page 55**. Methods for determining accuracy, precision, and completeness are also outlined in **Section 14, Page 55**.

Laboratory method detection limits (MDL) for Target Chemicals have been established (**Table 1**). The actual detection limits achieved for each sample analysis may be affected by chemical interference or sample dilution.

Implementing the procedures in the QAPP during data collection activities will allow a consistent quality of data to be maintained. This consistency will be accomplished through the formal standardization and documentation of field techniques and activities. All field activities will be planned in advance to ensure consistency with overall project objectives. Actual field and laboratory activities will be performed by properly trained and qualified personnel and will conform to specific procedures outlined in subsequent sections of this document. Project documents resulting from these activities will be reviewed for completeness, accuracy, and conformity with specific procedures.





## 5.0 SAMPLING PROCEDURES

Data collected during Phase 2A will be used to determine the distribution of Target Chemicals in groundwater, soil, and sediment. The procedures presented in this section are designed to ensure that all samples are collected in a manner consistent with project objectives, and are identified, preserved, and transported in such a manner that data are representative of the actual site conditions and no information is lost in sample transfer.

### 5.1 Types, Locations, and Numbers of Samples

The types, locations and numbers of samples to be collected are determined based on available data and are specified in Table 2. The locations and numbers of proposed groundwater monitor wells, soil borings, and sediment samples were selected to provide additional information on the horizontal and vertical distribution of Target Chemicals at and in the vicinity of the Montrose site.

Phase 2A field activities will include installation of a minimum of 18 off-site monitor wells and one on-site monitor well. Groundwater samples will be collected from all monitor wells shortly after pumps have been installed in the new wells. A second groundwater sampling round will be conducted approximately two weeks after the initial sampling. Additional groundwater sampling of these wells will be conducted quarterly for the following year to evaluate changes in water quality with time. Data from these rounds will be used to determine an appropriate schedule for future sampling rounds. Detailed methods for sampling groundwater are presented in Section 5.3.1, Page 13. Site-specific well locations, depths, and completion intervals are outlined in the SAP, Figures 5 through 9.

Soil samples will be collected on-site and from four areas south of the Montrose site. These four areas include, the SPRR right-of-way, the LADWP right-of-way, and an historical drainage on the Farmer Brothers Coffee



(Farmer Bros.) facility. The location, number of borings, number of samples per boring, and the depth intervals for each location are detailed in the SAP. Detailed soil sampling methods are presented in Section 5.3.2, Page 17 of this document.

Sediment samples will be collected from the Dominguez Channel in the vicinity of the Torrance Lateral discharge. Prior to sample collection, the thickness and distribution of sediment will be determined according to the procedures outlined in Section 6.5, Page 38. Sediment sample locations will be specified based on an evaluation of Part 2, Phase 1 sediment sample analytical results and the sediment thickness data to be obtained in the Phase 2A sediment survey. Criteria for selection of sediment sample locations are outlined in the SAP, Page 41.

On-site soil samples and all groundwater samples will be analyzed for the Target Chemicals; total DDT, total BHC, acetone, benzene, chlorobenzene, chloroform, 1,2-dichlorobenzene, 1,3-dichlorobenzene and 1,4-dichlorobenzene. The term total DDT refers to all DDT isomers and its metabolites DDD and DDE. The term total BHC refers to the alpha, beta, delta, and gamma BHC isomers. The Target Chemicals were selected by the EPA based on the results of previous on-site investigations conducted both by Hargis + Associates, Inc. and by the EPA contractor Metcalf & Eddy, Inc. In addition, groundwater samples collected from Phase 2A wells during the second sampling round will be analyzed for total dissolved solids, pH and common ions calcium, magnesium, sodium, potassium, alkalinity, fluoride, chloride, sulfate, nitrate, boron, and silica (Table 1).

Although not required under the Consent Order, common ion data are useful in assessing the hydrogeologic conditions in the vicinity of the Montrose site.

Off-site soil and sediment samples will be analyzed for total DDT and total BHC. Phase 1 analytical results indicate that total DDT and total BHC are the contaminants of concern in off-site soils and sediments. Analysis



for volatile organic compounds (VOC's) will be specified for off-site soil samples collected from the Normandie Avenue ditch only if the results of field screening with an organic vapor analyzer (OVA) indicate the possible presence of VOC's. The methods for OVA field tests are presented in **Section 6.3, Page 37**. Electrical conductivity (EC), pH, percent moisture, and total organic carbon (TOC) will be measured on selected soil and sediment samples to aid in screening remedial technologies (**Table 4**).

The proposed location of each sample is clearly defined in the SAP. The actual sample location and type of sample collected will be documented in a field notebook.

## **5.2 Monitor Well Installation**

Groundwater monitor well installation objectives are to obtain data on geologic conditions, provide water level information, and collect samples to analyze the chemical quality of the groundwater in the Bellflower aquitard and the Gage and Lynwood aquifers. These objectives will be achieved by conforming to standard operating procedures during monitor well design and construction, geologic logging, groundwater level measurement, and groundwater quality sampling. Quality assurance depends largely on field personnel adherence to standard operating procedures.

Prior to entering the field, personnel will contact applicable agencies and cities to ensure compliance with regulatory requirements regarding access, drilling, and well construction. Additionally, all field personnel will be familiar with the well construction details before well construction begins.

Upper Bellflower aquitard monitor wells will be constructed using a hollow stem auger drill rig. Bellflower sand, Gage and Lynwood aquifer monitor wells will be constructed using fluid rotary drilling methods. Only high grade bentonite type drilling muds will be used, and the mud must be



approved by the on-site geologist prior to use. Drilling fluid characteristics will be maintained through periodic monitoring of fluid weight, viscosity, and sand content.

Generalized design details for hollow stem auger and fluid rotary monitor wells have been summarized (**Figures 3 and 4**). Well screen and well seal depth intervals for the various monitor well types are proposed in detail in the SAP, **Figures 6 through 9**.

For fluid rotary well completions, mild steel conductor casing will be pressure grouted in place with neat cement to approximately 10 feet above the interval to be screened. Conductor casing will be installed to stabilize the borehole during well construction and to isolate the screened interval from the overlying units. The potential for cross-contamination is greatly reduced by isolating the overlying units and using fresh drilling fluid to complete the borehole below the conductor casing.

Stainless steel 316 or 316L well screen will be installed in all wells. In fluid rotary well completions approximately 10 feet of blank stainless steel well casing will be installed above the screen. All stainless steel well screen and casing will be steam cleaned prior to installation. Screen size and filter pack for all wells will be selected based on sieve analyses of samples representative of the hydrogeologic units to be screened. Blank PVC schedule 40 well casing will be installed from the top of the stainless steel casing or well screen to land surface. For fluid rotary well completions, centralizers will be placed at approximately 20-foot intervals along the well screen.

Filter pack will be installed in all wells from the bottom of the boring to approximately 2 feet above the screen. For hollow stem auger well completions, filter pack and seal materials will be placed by gravity by adding them directly into the annulus between the well casing and the auger stem. To minimize the possibility of the filter pack bridging between the well casing and the hollow stem auger, the filter pack will be installed in



2- to 3-foot increments as the auger is lifted out of the hole. For hollow stem auger monitor well completions, granular bentonite will be placed above the filter pack and hydrated with tap water. For fluid rotary well completions, the filter pack and bentonite seal materials will be installed with a 1- to 2-inch diameter tremie pipe. Approximately 3 feet of very fine sand will be placed in the annulus above the granular bentonite seal in hollow stem auger completions and above the filter pack in fluid rotary well completions. The very fine sand will act as a grout filter to reduce the possibility of grout entering the filter pack.

A well seal will be installed from the top of the grout filter to land surface. The well seal in the hollow stem auger well completions will consist of a sand-cement slurry. The slurry will have a minimum of seven sacks of neat cement per cubic yard. The slurry will be mixed with 5 to 6 gallons of water per sack of neat cement. The well seal in fluid rotary well completions will consist of a high solids bentonite grout such as NL Baroid's Benseal or American Colloid's Volclay Grout. The grout will be mixed following manufacturers' recommendations.

Dedicated gas-activated bladder pumps made of teflon and stainless steel will be installed in each monitor well. In upper Bellflower aquitard monitor wells the bladder pump will be installed near the bottom of the screen. In deeper wells the bladder pump will be set immediately above the well screen inside the blank stainless steel well casing. Dedicated electric submersible pumps will be installed above the bladder pumps in deeper wells, to allow efficient purging of the well prior to sampling. Purge pump depth will be determined based on well performance during development. Upper Bellflower aquitard monitor wells may be equipped with a gas-driven purge pump depending on well performance during development. The purge pump and teflon bladder sampling pump installations and configurations are specifically designed to enable efficient purging of groundwater from monitor wells to eliminate the influence of stagnant water and to minimize groundwater turbulence in the vicinity of the sample pump intake. This design has been



implemented to address the concern that VOC's may escape during well purging and sampling activities.

The drilling and completion of each monitor well will be overseen by a geologist/hydrogeologist responsible for description of lithology, selection of screened intervals, and determination of final well depth. Drill cuttings will be collected frequently during the drilling of all fluid rotary boreholes. Hollow stem auger monitor well borings will be logged by inspecting auger cuttings and soil samples collected from drive samples and core barrels.

Sieve analyses were conducted on undisturbed lithologic samples from the upper Bellflower aquitard, Bellflower sand, and Gage aquifer, collected from exploratory borings EB-1 and EB-2 during the Part 2, Phase 1 tasks. Well screen slot sizes for Phase 1 monitor wells were selected based on these sieve analyses. Based on the lithology reported at the Del Amo site exploratory boring, it is anticipated that the same well screen slot size will be appropriate for Phase 2A monitor well installations. This will be verified by conducting sieve analyses on cutting samples from each of the hydrogeologic units to be screened.

Decontamination procedures for monitor well drilling will require that downhole hollow stem auger drilling equipment be steam cleaned prior to installation of each upper Bellflower monitor well. Downhole fluid rotary drilling equipment will be steam cleaned prior to installation of each well cluster. After the conductor casing is installed in each Bellflower sand monitor well, new mud will be mixed and the interval to be screened will be drilled. The same drilling mud will be used to drill the same lithologic interval for the conductor casing boring of the adjacent Gage aquifer monitor well. After the conductor casing is installed in the Gage aquifer monitor well, new mud will be mixed again. Using new mud for each hydrogeologic unit will avoid potential cross-contamination by drilling equipment between monitor wells at a given well cluster.



Following completion of each phase of monitor well installation, the measuring points of each new well will be surveyed to a vertical precision of 0.01 foot and an horizontal precision of 0.1 foot.

### 5.3 Sampling Techniques

Samples collected during Phase 2A will include groundwater samples collected from existing on-site monitor wells and Phase 2A on- and off-site monitor wells, soil samples collected from on-site and off-site borings, and sediment samples collected from the Dominguez Channel (Table 2). Specific procedures for collecting groundwater, soil, and sediment samples are outlined below.

#### 5.3.1 Groundwater Sample Collection

Groundwater samples will be collected from existing and newly constructed monitor wells. Prior to sample collection, the wells will be pumped until approximately three casing volumes of water are removed. The electrical conductance (EC), pH, and temperature of the well discharge will be measured to ensure that these parameters have stabilized prior to sample collection.

EPA split, duplicate and laboratory check samples will be collected and field blank samples prepared during each sampling round (Table 3). Trip blanks will be shipped with water samples for VOC analysis. Trip blanks will consist of water samples containing Laboratory Type II Reagent Grade Water, which will be prepared in the laboratory prior to sample collection, shipped with the empty sample containers, labeled, and returned to the laboratory for analysis. Field blank water samples will also consist of Laboratory Type II Reagent Grade Water but will be prepared in the field at predetermined sample locations. Duplicate water samples and laboratory check samples will consist of additional groundwater samples collected from predetermined sample locations. These locations were selected to provide a



representative population of samples from the new monitor well installations. Brown and Caldwell Laboratory, Pasadena, California, will be the primary analytical laboratory for sample analysis. Analytical Technologies, Inc., San Diego, California will be used for analysis of laboratory check samples. All groundwater sampling equipment including purge pumps, sample pumps and sample containers are dedicated for each monitor well. Because no decontamination procedures between sample locations are required, rinsate samples from groundwater sampling equipment are not necessary.

The following procedures will be used to obtain groundwater samples.

A. BEFORE ENTERING THE FIELD, HARGIS + ASSOCIATES, INC. PERSONNEL WILL:

- .. Review project objectives, sampling locations, schedule, and required analyses.
- .. Review sampling procedures, preservation methods, packing and shipping procedures.
- .. Notify the EPA and its contractors at least 20 days before sampling to allow mobilization for collecting sample splits.
- .. Review health and safety procedures.
- .. Review appropriate permits and site access procedures.

B. IN THE FIELD, HARGIS + ASSOCIATES, INC. PERSONNEL WILL:

- .. Review the locations for duplicate and laboratory check sample collection and field blank sample preparation, prior to commencing field work each day.
- .. Record daily weather conditions and site characteristics.





- .. Measure depth to water in each well with steel tape, electric sounder or both.
- .. Record OVA measurements at each wellhead, in the well vault, and in the field personnel's breathing zone.
- .. Calculate the casing water volume for well evacuation purposes.
- .. Pump each well until approximately three casing volumes of water have been evacuated, or until the EC, pH and temperature have stabilized.
- .. Record the following information in the field notebook:
  - static water level
  - time that pumping begins
  - time of sample collection
  - pump discharge rate using volumetric calculation
  - field parameters, including EC, pH, and temperature
  - time that pumping or bailing stops
  - OVA measurements of field personnel's breathing space during well purging or sampling.
- .. Rinse nonpreserved sample containers with discharge water to ensure that any possible contaminants in the sample bottle are removed. Pretreated sample containers, such as those for pesticides, shall not be rinsed with sample water. Bottle preparation is discussed in Section 5.4.1, Page 24.
- .. Collect water samples for pesticide analysis in two 1-liter amber glass bottles with teflon-lined screw cap lids. Collect water samples for common ion analysis in 1-liter polyethylene bottles. Collect water samples for nitrate analysis in 100-milliliter (ml) polyethylene bottles pretreated with sulfuric acid.



- .. Reduce the bladder pump discharge to a gentle flow prior to collecting water samples for VOC analyses.
- .. Collect water samples for VOC analysis in two 40-ml glass vials with teflon-lined caps. To avoid aeration, the glass vial will be held at an angle so that the stream of water flows down the side. The vial will be filled until it overflows to eliminate any air bubbles, then the teflon-lined cap will be replaced. Two vials will be collected for each sample. When sampling for VOC's, the 40-ml sample vials will have zero head space after being filled and capped. The vial will be turned upside down and tapped to check for air bubbles. If there are any bubbles, the vial will be emptied, refilled, and recapped, then checked for air bubbles again. This procedure will be repeated until an acceptable sample is collected. An acceptable sample is one with no observable air bubbles.
- .. Include trip blank water samples for VOC analysis with each shipping container containing field VOC water samples. Label the laboratory-prepared trip blank water samples in the field and submit them to the laboratory as blind samples.
- .. Prepare field blank water samples for pesticide and VOC analyses at the predetermined monitor well locations and submit them to the laboratory as blind samples. Ship field blanks with samples each day groundwater samples are collected.
- .. Collect one duplicate and one laboratory check sample for every 10 samples collected, or a minimum of one duplicate sample per sampling day. These groundwater samples will be analyzed for the Target Chemicals.



- .. Label each sample container with project number, well number, pre-determined sample number, collector's name and company, and the analyses to be performed (Appendix A).
- .. Record all pertinent data concerning each sample in the field notebook.
- .. Prepare letters of transmittal, chain-of-custody documentation, and laboratory schedules for analyses to be performed at the end of each sampling event (Section 7).
- .. Package the sample according to the procedures outlined in Section 5.4, Page 24.
- .. Place samples on ice as outlined in Section 5.4, Page 24 and transport to the laboratory within 24 hours of collection.

### 5.3.2 Soil Sample Collection

Soil samples will be collected using split-tube samplers, continuous core samplers, or small hand-driven samplers, all fitted with new brass sampling tubes. Soil borings will be advanced using a hollow stem auger rig or a hand auger.

The following procedure will be used to collect soil samples:

#### A. BEFORE ENTERING THE FIELD, HARGIS + ASSOCIATES, INC. PERSONNEL WILL:

- .. Review project objectives and identify sites to be drilled; review sampling locations and schedule, sampling equipment and supplies, and required analyses.



- .. Notify EPA and its contractors at least 20 days before sampling to allow mobilization for collecting sample splits.
- .. Review health and safety procedures with all personnel.
- .. Review appropriate permits and site access procedures.

B. IN THE FIELD, HARGIS + ASSOCIATES, INC. PERSONNEL WILL:

- .. Review duplicate soil sample locations prior to commencing field work each day.
- .. Cover ground surface around the immediate borehole site with plastic sheeting to prevent mixing of surface and subsurface soil.
- .. Decontaminate 6-inch brass tubes and soil sampler prior to sampling. Place 6-inch brass sample tubes into the sampler body. Label top and bottom of tubes. Attach sampler to the hammer or drill rig and drive or core the sampler into the soil ahead of the boring.
- .. Carefully remove the sample tubes after the sampler is retrieved. When possible the middle tube will be used for analyses. The other tubes will be used for splits, duplicates, or sample analysis if sample recovery from the middle tube is insufficient. When sampling at 1-foot intervals with a 5-foot core sampler, every other tube will be submitted for analysis.
- .. Cover sample tube ends with teflon liners and plastic end caps immediately upon retrieval. The end caps will be secured with electrical tape.
- .. Record OVA measurements from soil obtained from each drive sampler collected from the Normandie Avenue ditch (Section 6.3, Page 37).



- .. Label each sample with project number, hole number, depth interval, predetermined sample number, date and time of sampling, collector's name and company, and the analyses to be performed (Appendix A). Note the end of the sample to be analyzed on each sample tube. Place each sample in a plastic bag and immediately store in an ice chest.
- .. Per EPA request, analyze adjacent sampling tubes as theoretical duplicate samples for one out of every 10 soil samples (Section 11.2.1, Page 48).
- .. Label, package, and store duplicate samples following the same procedures used for the primary samples. The identity of duplicate samples will not be known to the laboratory.
- .. Describe soil encountered in the field notebook, including color, texture, mineral composition, moisture content, grain size, shape, sorting, and degree of induration.
- .. Enter the following information into a field notebook each time a soil sample is collected:
  - date of sample collection
  - hole location and number
  - depth interval
  - description of the soil
  - time of sample collection
  - OVA readings
  - any other pertinent information, including any difficulties in sampling or unusual drilling characteristics.
- .. Implement the following decontamination procedures for nondedicated samplers used to collect samples for laboratory analyses:
  - clean with nonphosphate detergent wash
  - rinse with tap water



- rinse with reagent grade methanol or hexane
  - rinse twice with distilled water.
- .. Collect sampling equipment rinsate samples at a rate of one per day during sampling decontamination. The rinsate will consist of Laboratory Type II Reagent Grade Water collected in a rinsate sample container directly from the sampling equipment. Rinsate samples collected during on-site soil sampling will be analyzed for Target Chemicals. Rinsate samples collected from off-site soil sampling will be analyzed for nonvolatile Target Chemicals. One 1-liter amber glass bottle and one 40-ml glass vial, each with teflon-lined screw caps, will be submitted. Sample labeling, packaging and handling procedures are described in Section 5.3.1, Page 17. Wash water will be stored at the Montrose site until results of soil analysis are available. The wash water will be disposed of in an appropriate manner depending on analytical results. Disposal is discussed further in Section 5.6, Page 29.
- .. Complete chain-of-custody forms, laboratory analysis schedules, and appropriate transmittal letters, as outlined in Section 7, Page 39.
- .. Package samples according to the procedures outlined in Section 5.4, Page 24, and deliver to the laboratory within 24 hours of collection.

### 5.3.3 Sediment Sample Collection

Sample collection procedures will vary depending on the sediment sampling method used. Alternate sediment sampling methods are discussed in the SAP, Page 44. If samples for laboratory analysis are collected with the Vibra Corer™ device, brass or stainless steel inner tubes will line the drill stem. If necessary, the tubing will be secured with a pipe vise and sectioned



into discrete samples no more than 6 inches in length using a pipe cutter. The top and bottom of each discrete sample will be noted before sectioning the tube. Before the tubes are cut, the open tube ends will be sealed with teflon liners and plastic end caps. End caps will be secured with electrical tape. EPA split and duplicate samples will consist of the core samples collected immediately above or below the primary core sample.

If the thin-wall suction sampler is used, discrete sediment samples not exceeding 6 inches in length will be extruded with the plunger. Sediment will be extruded directly into 1/2-liter wide mouth glass jars equipped with teflon-lined threaded caps. EPA split or duplicate samples will consist of the core samples immediately above or below the primary core sample.

If the hand augering technique is used, sediment will be transferred with a decontaminated utensil from the auger to a 1/2-liter wide mouth glass jar. When sediment thickness is great enough to require sample collection at discrete depth intervals, a length of decontaminated 3- to 4-inch I.D. thin-wall temporary conductor casing consisting of PVC, sheet metal or suitable alternate will be pushed into the sediments to stabilize the sediment deposit and borehole. The hand auger will be inserted inside the casing and advanced at 6-inch intervals to collect the discrete samples. EPA split and duplicate samples will be collected by alternately transferring sediment from the auger into two sample containers.

A background sediment sample location has been selected at the upstream end of the clay-lined portion of Dominguez Channel. The background sediment sample location is described in the SAP, Page 42. Background sediment samples will be collected at 2-foot intervals to the bottom of the sediment deposit.

Because of the potential complexity of obtaining sediment samples at depth, it is possible that only two or three samples per day may be collected. Duplicate sediment samples will not be collected on a one per day minimum basis because the daily production of samples may be very low and because duplicate sediment samples are not true duplicates and are therefore of



limited use. Duplicate samples will be submitted to the lab at an overall rate of 10 percent of the total number of sediment samples collected, by selecting one of every 10 consecutive samples for duplicate analysis.

Decontamination procedures for sampling devices that will be reused will follow the protocol described in Section 5.3.2, Page 19. Each sample will be sent to the laboratory within 24 hours after sample collection.

The following procedures will be used to obtain sediment samples:

A. BEFORE ENTERING THE FIELD, HARGIS + ASSOCIATES, INC. PERSONNEL WILL:

- .. Review project objectives; review sampling locations and schedule, sampling equipment and supplies, and required analyses.
- .. Notify EPA and its contractors for purposes of collecting sample splits.
- .. Review health and safety procedures with all personnel.
- .. Review appropriate permits and site access procedures.

B. IN THE FIELD, HARGIS + ASSOCIATES, INC. PERSONNEL WILL:

- .. Review duplicate sediment sample locations prior to commencing field work each day.
- .. Collect each sediment sample according to one of the three alternate sediment sampling methods discussed in this QAPP.
- .. Label each sample with project number, hole number, depth interval, predetermined sample number, date and time of sampling, collector's name and company, and the analyses to be performed (Appendix A). Note the end of the sample to be analyzed on each sample tube.





Place each sample in a plastic bag and immediately store in an ice chest.

- .. Label, package, and store duplicate samples following the same procedures used for the primary samples. The identity of duplicate samples will not be known to the laboratory.
- .. Describe in the field book sediment encountered, including color, texture, mineral composition, grain size, shape, sorting, and degree of induration.
- .. Enter the following information into a field notebook each time a sediment sample is collected:
  - date of sample collection
  - sample location and number
  - depth interval
  - description of the sediment
  - time of sample collection
  - any other pertinent information, including any difficulties in sampling or unusual drilling characteristics.
- .. Decontaminate nondedicated sediment samplers used to collect samples for chemical analyses following the protocol described in Section 5.3.2, Page 19.
- .. Complete chain-of-custody forms, laboratory analysis schedules, and appropriate transmittal letters as outlined in Section 7, Page 39.
- .. Package samples according to the procedures outlined in Section 5.4, Page 24 and deliver to the laboratory within 24 hours of collection.



## 5.4 Sample Handling, Packaging, and Shipping

This section specifies the sample handling, packaging, and shipping procedures that will be followed by the investigative team. Types and sizes of containers to be used, container preparation, recommended volume of sample to be collected, sample preservation, and maximum laboratory holding time for groundwater samples under EPA guidelines have been summarized (Table 4). Letters of transmittal, chain-of-custody records, and laboratory schedules will be completed, packed in a waterproof bag, and included with the samples. Samples must be packed in plastic air bubble packing material to avoid breakage or contamination. All samples will be stored on ice immediately after sample collection and identification.

Samples will be transported to the laboratory by courier, by a laboratory representative, or by Hargis + Associates, Inc. personnel. In the event that a courier is used, custody tape will be placed on all samples and on the shipping containers. Hargis + Associates, Inc. will arrange in advance for Saturday delivery to allow for receipt of samples by the laboratory within 24 hours after sample collection. A sufficient quantity of ice will be placed in the shipping container to maintain a temperature of 4°C until the container is received by the laboratory.

### 5.4.1 Groundwater Samples

Water samples for analysis of VOC's will be collected from each monitor well in two 40-ml glass vials equipped with teflon-backed silicon septum screw caps. Correctly prepared bottles and septa are washed with detergent, rinsed with distilled water, and dried for one hour at 105°C. The 40-ml glass vials will be rinsed with sample water before the sample is collected.

Each VOA vial will be completely filled with water. Zero head space in each vial will be ensured by inverting the vial and gently tapping on the



cap. If air bubbles appear the vial will be discarded and the process repeated until zero head space is confirmed.

Groundwater samples collected for analysis of total DDT and total BHC will be collected in two 1-liter amber-colored glass bottles sealed with teflon-lined caps. Bottles will be filled directly and will not be rinsed with sample water. New bottles and liners prepared in the laboratory will be rinsed with methylene chloride and dried by vacuum or other safe means until no solvent remains.

Water samples for common ion analyses will be collected in clean 1-liter polyethylene bottles. Water samples for nitrate analysis will be collected in 100-ml polyethylene bottles pretreated with sulfuric acid. Containers for all analyses will be cleaned and prepared in the laboratory as specified in the EPA-approved method for each type of analysis. All water samples will be taped, labeled, and stored at 4°C by placing on blue ice or double-bagged ice in plastic ice chests until delivered to the laboratory. Once the samples are logged in at the laboratory, the samples are stored at 4°C in a refrigerated locker.

Primary, duplicate, EPA split and laboratory check samples will be collected concurrently for VOC and pesticide analyses. The water samples will be collected by alternately filling a series of sample containers at the wellhead from the bladder pump discharge.

Provisions for split samples from the monitor wells will be made. Split samples will be provided to the EPA in the field. EPA and its contractors will be notified at least 20 days prior to commencement of field sampling to allow mobilization of their sampling team and selection of a laboratory. Coordination with EPA and its contractors will be maintained throughout the groundwater sampling process.



#### **5.4.2 Soil Samples**

Soil samples will be collected in brass sampling tubes which will line all the soil sampling devices to be used in this study. Immediately after sample collection, the tubes will be capped, labeled, and packed for shipping. All sampling tubes will be wrapped individually in plastic bags and placed in an ice chest. Whenever possible the middle tube will be used for analyses unless sample recovery is insufficient. Procedures for collecting samples and capping and labeling tubes are described in Section 5.3.2, Page 17.

Split soil samples will be provided to the EPA in the field. Split and duplicate samples will be handled, preserved, and stored in a manner identical to the primary sample. Coordination with EPA and its contractors will be maintained throughout the sampling process.

#### **5.4.3 Sediment Samples**

Sediment samples will either be transferred into two 1/2-liter wide mouth glass jars with teflon-lined threaded caps or sealed in the inner tubes that line the drill stem of the Vibra Corer™ device. The inner tubes will be made either of stainless steel or of brass and will be decontaminated prior to use. Decontamination procedures are outlined in Section 5.3.2, Page 19. Sediment samples will represent no more than a 6-inch vertical interval of the sediment deposit. All sample containers will be individually wrapped and stored on ice in an ice chest.

#### **5.4.4 Methods for Particle Size Analysis**

Particle size sieve analyses will be conducted on two sample matrices; soil and water. The purpose of the sediment particle size analysis is to evaluate the grain size distribution of Dominguez Channel sediments. A



representative Dominguez Channel sediment sample will be collected for particle size analysis as discussed in the SAP, Page 40. No chemical analyses will be conducted on sediment collected for particle size analyses. The purpose of the soil particle size analyses is to evaluate the relationship between particle size and DDT concentration in soil from the Normandie Avenue ditch. Two soil samples from boring T52 will be sieved and the retained soil analyzed for Target Chemical pesticides by EPA Method 8080. Sieve work for both sample materials will follow the standard method for particle size analysis of soil and sediment as outlined by the American Society for Testing and Materials (ASTM, 1986).

The contents of the brass liner will be emptied onto a decontaminated metal tray and baked in a portable oven at approximately 38°C until all residual moisture has been removed. The soil will be transferred to a stainless steel bowl and ground with a pestle to break up the soil aggregate into separate grains. Approximately 150 grams of soil per sample will be selected by quartering the total soil volume. The dry sample will be weighed to the nearest 0.1 gram with a triple beam balance.

The expected sand size will range from medium- to very fine-grained based on the particle-size distribution observed in soil samples collected from soil boring T52. Square mesh woven-wire No. 10, No. 40, and No. 200 cloth sieves will be used to determine the range of particle sizes. The soil retained on each sieve mesh and the pan will be weighed to the nearest 0.1 gram with a triple beam balance.

After the retained fractions have been weighed, the individual particle-size fraction retained on each sieve will be rinsed with distilled water to remove silt and fine sediment which may be attached to the sand grains. The rinse water from all size fractions of a sample will be composited in a 1-liter amber glass jar. The rinse water sample will be filtered in the lab through a 0.45-micron filter and analyzed by EPA Method 608 to evaluate any removal of DDT due to rinsing. Each washed particle-size fraction will be



transferred to a separate 8-ounce glass jar with a teflon-lined screw cap lid and analyzed by EPA Method 8080.

Equipment used for particle size analysis will be decontaminated prior to and between sieve analyses. The soil tray, bowl, pestle, transfer funnel, and sieves will be decontaminated following the procedures described in Section 5.3.2, Page 19. An equipment rinsate sample will be collected from the sieve equipment after decontamination procedures have been completed. The equipment rinsate sample will be collected after the first soil sample is sieved but prior to sieving the second soil sample. The equipment rinsate sample will be collected in one or more 1-liter amber glass jars and analyzed by EPA Method 608.

Commonly, residual sand grains become trapped between the sieve mesh after the particle size fraction has been transferred out of the sieve. In order to avoid cross-contamination between sieve samples, the sieves will be baked in a portable oven at low temperature. The heat will expand the sieve material and allow residual particles to be gently removed by brushing.

The sieve analyses will be conducted in the field the day following sample collection. Therefore, the sieved samples cannot be submitted to the laboratory within 24 hours of sample collection. To ensure that holding times for soil and water are not exceeded, Brown and Caldwell Analytical Laboratories will be notified on the day following sample collection that six to eight additional samples with a reduced extraction holding time will be submitted by Hargis + Associates, Inc. personnel.

## 5.5 Sampling Safety and Decontamination Considerations

An OVA will be utilized during drilling and sampling activities for detection of volatile organic emissions from the borehole or monitor well being drilled. If VOC's are detected, appropriate safety actions will be taken in accordance with the specifications of the health and safety plan



(HSP) (Clayton Environmental Consultants, Inc., 1988). Additional site safety requirements including decontamination of personnel and protective equipment are covered in the HSP. Procedures for disposal of decontamination fluids and drilling cuttings are discussed below.

## **5.6 Disposal of Investigation-Derived Material**

Potentially hazardous materials generated from the remedial investigation can be categorized into four general groups: water, drilling fluids, auger cuttings, and solid waste. Water will be generated during monitor well development and purging as well as during decontamination activities. Bentonite drilling fluids will be generated from exploratory boring and monitor well construction activities. Auger cuttings will be generated from soil sampling and monitor well construction activity. Solid waste comprises used health and safety equipment such as Tyvek suits, protective gloves, and plastic sheets. Disposal options for the various waste groups are discussed in the following sections. The EPA will be notified of Montrose's final disposal plan prior to actual disposal. A volumetric estimate of the disposable materials to be generated during the remedial investigation and a summary of the temporary storage and final disposal options has been prepared (Table 5).

### **5.6.1 Water**

Montrose is currently investigating the procedures and requirements necessary for disposal of waste water. Three waste water disposal options are under consideration: 1) transportation to an off-site water treatment facility; 2) on-site treatment and subsequent discharge to the storm sewer; and 3) on-site evaporation. On-site treatment or evaporation may require approval from the South Coast Air Quality Management District (SCAQMD). Discharge of treated waste water to the storm sewer would require a National Pollutant Discharge Elimination System (NPDES) permit from the Los Angeles



Regional Water Quality Control Board (LARWQCB). Ongoing discussions and correspondence with waste disposal firms, water treatment plant operators, and the EPA during the drafting and reviewing of this plan will determine final water disposal options.

### 5.6.2 Drilling Fluid and Cuttings

If practical, drilling mud will be segregated into drilling fluid and drill cuttings and disposed of separately. Drilling fluid will be transferred by vacuum truck from the well site to large capacity Baker Tanks for temporary on-site storage. Cuttings will be contained in 55-gallon drums or roll-off bins for temporary on-site storage. When a Baker Tank or roll-off bin has been filled to capacity, representative samples will be collected and sent to the laboratory for composite analyses. Analyses will include pesticides, VOC's, metals, pH and electrical conductivity.

If the analytical results indicate that chemical concentrations are below Soluble Threshold Limit Concentrations (STLC) listed by the State of California (1985), the drilling fluid or cuttings will be disposed of as nonhazardous waste at a Class II landfill such as the Los Angeles County Sanitation District's (LACSD) Puente Hills facility or the Liquid Waste Management, Inc. facility near McKittrick, California. If the drilling fluid is determined to be hazardous it will either be disposed of at a Class I landfill such as the Chemical Waste Management, Inc. facility near Kettleman City, California, or it will be stored on-site until on-site remedial methods are determined. If the drilling fluid is determined to be hazardous, on-site solidification of the drilling fluid will be considered in order to reduce the cost of the waste disposal. If the drill cuttings are determined to be hazardous, they will either be disposed of at a Class I landfill such as the Casmalia Resources facility near Santa Maria, California, or they will be stored on-site until on-site remedial methods are determined.

Non-haz.





To obtain a representative sample from a Baker tank, a three-dimensional sampling strategy will be used. The sampling strategy will consist of dividing the top surface of the tank contents into a hypothetical grid. The depth of the tank will be divided into hypothetical layers. The thickness of each layer will be equal to or larger than the length of the sampling device. Four samples will be collected at random grid points. Logistical considerations will determine the selection of the actual sampling device to be used. Possible sampling devices include Coliwas tubes, PVC bailers, or weighted bottles. Each sample will be identified by tank number and grid point coordinates. If the drilling mud waste stream is contained in more than one tank, samples will be composited in the laboratory and a total of four analyses will be performed. The discrete samples will be retained in the laboratory in case analytical results indicate the need for discrete analyses or recompositing of samples. A similar methodology will be used to obtain representative samples of drill cuttings stored in roll-off bins.

#### 5.6.3 Auger Cuttings

Auger cuttings from off-site and on-site soil and monitor well borings will be collected in 55-gallon drums and roll-off bins. Auger cuttings will be initially segregated by borehole and depth interval in order to minimize the volume of potentially hazardous materials requiring disposal. Disposal of soil boring cuttings will be based on analytical results from soil samples collected for the remedial investigation. Disposal of augered monitor well cuttings will be based on laboratory analysis of a composite sample. Auger cuttings determined to be nonhazardous will be disposed of at a Class II landfill. If determined to be hazardous, auger cuttings will be disposed of at a Class I landfill or will be stored on-site until appropriate soil remediation methods are determined.



#### **5.6.4 Solid Waste**

Tyvek suits, gloves, Visqueen sheets and other disposable field equipment used in the exclusion zone will be contained in 55-gallon drums or roll-off bins and disposed of at a Class I landfill.

#### **5.7 Borehole Abandonment**

Borehole abandonment procedures will vary depending upon the total depth of the borehole to be abandoned. Auger borings completed above the water table will be backfilled with a neat cement or a sand-cement slurry. Boreholes completed below the water table will be abandoned with neat cement or a sand-cement slurry to be tremied from the bottom of the boring to land surface. The neat cement or sand-cement slurry will be mixed using 5 to 6 gallons of water per sack of cement. A minimum of seven sacks of cement per cubic yard will be used for sand-cement slurry mixtures.



## 6.0 FIELD MEASUREMENTS

This section describes the routine procedures to be followed by personnel performing field measurements. Field measurements include, but are not limited to, borehole logging, water level measurements, electrical conductivity, temperature, pH, well discharge measurements, and OVA soil sample screening. Field measurements will be recorded on the appropriate forms (Appendix B).

### 6.1 Borehole Logging

During the drilling of exploratory borings, monitor wells and soil borings, a complete log of the conditions encountered during drilling will be maintained. This includes lithologic and hydrogeologic descriptions along with notations on drilling particulars. All logging will be supervised by a qualified geologist/hydrogeologist.

An accurate lithologic log will be recorded by following these guidelines:

- .. For 60-foot deep soil borings, split tube drive samples will be collected at 5-foot intervals between approximately 20 and 60 feet below land surface (bls) for lithologic control.
- .. Soil samples for lithologic descriptions will be collected at the direction of the field geologist. For hollow stem auger monitor wells, the borings will be logged by inspecting auger cuttings, and soil samples will be obtained from drive samples and core barrel.
- .. Moisture content of all auger samples will be noted as well as the depth at which groundwater is first encountered if apparent.



- .. Drive sample blow counts will be recorded.
- .. Fluid rotary cuttings will be collected frequently by placing a fine mesh screen at the borehole fluid discharge point.
- .. Drilling speed and rig behavior will be noted to help verify the nature of the material encountered.
- .. Drilling fluid properties will be monitored and controlled.
- .. The description of the drill cuttings will include, when distinguishable in the field, the following:
  - color of cuttings
  - grain size, shape and sorting
  - moisture content
  - mineral composition
  - descriptive comments, such as degree of cementation, staining
- .. The on-site geologist/hydrogeologist will be responsible for recording all the above information on a lithologic log sheet in the field notebook (Appendix B).

## 6.2 Water Level Measurements

Water levels will be measured with an electric sounder or steel tape. The electric sounder used for water level measurements will have marks on the sounder line at regular intervals such as 1, 5, 10, or 25 feet. Electric sounders will be calibrated periodically to maintain accuracy as described in Section 8.0, Page 44.

Steel tapes will be checked for kinks and will have the first 1 to 10 feet chalked before each measurement.



The following procedures will be used for measuring water levels:

A) BEFORE ENTERING THE FIELD, HARGIS + ASSOCIATES, INC. PERSONNEL WILL:

- .. Identify the wells to be measured.
- .. Identify established measuring point for each well.

B) IN THE FIELD, HARGIS + ASSOCIATES, INC. PERSONNEL WILL:

- .. Sound each well twice for depth to water to the nearest 0.01 foot. When using a steel tape the variation must be no more than 0.05 feet between the two measurements. When using an electric sounder the variation must be no more than 0.1 feet between the two measurements. When using a steel tape, lower slowly to minimize contact with casing.
- .. Identify the measuring point at the wellhead. The same measuring point must be used for subsequent measurements.
- .. Record information in the field notebook.
- .. Decontaminate equipment with a nonphosphate detergent wash and a tap water rinse before proceeding to next well.

### 6.3 Electrical Conductivity, pH, Temperature and Organic Vapor Measurements

An electrical conductivity (EC) meter, a pH meter, and a field thermometer will be used for measuring the field parameters EC, pH, and temperature in water samples.

Prior to each use, the probes on the EC meter and the pH meter will be thoroughly rinsed with distilled water, and the pH meter will be calibrated



in known pH buffer solutions. Adjustment for temperature differences between the buffer solution and the sample will be made. All manufacturer's instructions for use of the instruments will be followed.

A representative water sample will be placed in a glass transfer bottle, or measurements will be made directly at the well discharge point. Measurements will be made as follows:

- .. Rinse the transfer bottle with sample water prior to filling.
- .. Submerge the probe in the transfer bottle immediately and take measurements accordingly.
- .. Record field measurements in a field notebook along with the sample I.D., sample location, the time and date of measurement.
- .. Do not use the water in the transfer bottle for laboratory analysis.
- .. If recalibration indicates problems with instrument probes, change probes and repeat the procedures for recalibration.

During all drilling operations, an OVA will be routinely used for detection of VOC emissions. During soil sampling operations, an OVA will be utilized to monitor field personnel's breathing zone for safety purposes and to detect VOC's in Normandie Avenue ditch soil samples.

The field VOC screening procedure will be conducted to determine whether Normandie Avenue ditch soil samples will be analyzed for VOC's by EPA Method 8240. Results of the field test will be used for screening purposes only and are not intended to quantify VOC concentrations in the soil. Soil samples will be submitted to the laboratory for EPA Method 8240 analysis if the results of the head space screening procedure indicate the presence of VOC's at concentrations greater than 5 ppm above background. The Foxboro OVA Model 128 has a minimum detectable limit for methane of 0.2 ppm and an

*Field screening  
Only for  
Normandie  
Ave.  
ditch*



accuracy of  $\pm 20$  percent. Only calibrated instruments will be used for screening soil samples. A backup OVA will be available during the soil screening activity. Background organic vapor levels in the ambient air will be measured and recorded in the field notebook. The screening procedure will be performed on the most sensitive scale on the OVA.

The entire contents of a soil sampling tube will be removed from the tube and immediately placed in a 1-gallon ziplock bag. The soil will be crumbled into small pieces and allowed to stand in the bag for approximately 15 minutes. After 15 minutes, the ziplock bag will be pierced with the OVA probe and a reading will be recorded in the field notebook. If OVA readings for methane in soil collected from Normandie Avenue ditch are greater than 5 ppm above background, an EPA Method 8240 analysis will also be specified for the associated sample being submitted to the laboratory. For health and safety purposes, VOC readings in the exclusion zone air space and in background areas will be recorded. If VOC's are detected, appropriate safety actions will be taken in accordance with the HSP. Exposure limits for selected contaminants have been summarized (Table 6).

#### 6.4 Well Discharge Measurements

During pumping for groundwater sampling, measurements will be made to determine the flow rate of the water discharging from the well as follows:

- .. Use 5- to 55-gallon container and a stopwatch to measure well discharge.
- .. Direct well discharge into the container, and start the stopwatch simultaneously.
- .. Turn the stopwatch off when the container is full.



- .. Calculate the rate of discharge by dividing the container volume in gallons by the time required to fill the container in minutes. Record the time of measurement and the calculated rate of flow in gallons per minute (gpm).

Data forms for this field measurement activity have been included (Appendix B).

### 6.5 Sediment Thickness Survey

The width and thickness of sediment deposits in Dominguez Channel will be estimated by collecting sediment thickness information from 3-point transects across the channel. The location and number of transects are presented in the SAP, Figure 11.

Sediment thickness will be measured directly from cores obtained from either of the two coring methods described in the SAP, Page 39. If sediment thickness cannot be directly measured from core samples, the depth to the channel liner will be determined by hand augering through the sediment to the clay liner. The depth from the water surface to the top of the sediment and the depth from the water surface to the channel liner will be measured and recorded.

It has been determined through test sampling that the clay liner is very dense and cohesive and is much more resistant to penetration than the overlying sediment. Variations in penetration resistance will be noted and the penetrated material will be visually inspected to ensure that the integrity of the channel lining is maintained during the sediment thickness survey and sediment sampling.





## 7.0 SAMPLE CONTROL/CHAIN-OF-CUSTODY

This section describes standard operating procedures for sample identification and chain-of-custody. The purpose of these procedures is to ensure maintenance of accurate documentation during sample collection, transportation, and storage prior to analysis.

### 7.1 Standard Operating Procedures

Sample identification documents must be carefully prepared so that identification and chain-of-custody can be maintained and sample disposition can be controlled. The sample identification documents that will be used as part of the Phase 2A investigation are prepared sample labels, chain-of-custody record/analytical services requests, sample transmittal letters, and field notebooks (Appendix A).

Preprinted and prenumbered adhesive sample labels must be secured to the sample containers by the sampler. Sample documentation forms and labels will be filled out in ink. Where necessary, the label will be protected from water and solvents with clear tape.

Sample documentation forms will include the following information:

- sample number
- project number
- sample site name
- sampling date
- sampling personnel
- shipping method and date
- sample description
- sample matrix and estimated concentration range
- sample volume and number of containers
- sample destination



- preservatives used
- analyses required
- special handling procedures

## **7.2 Chain-of-Custody**

Official custody of samples must be maintained and documented from the time of sample collection up to the validation of analytical results. To document sample possession, chain-of-custody procedures are followed as outlined in the sections below.

### **7.2.1 Field Custody Procedures**

To the extent possible, the quantity and types of samples and sample locations are determined before initiating actual field work. As few people as possible should handle samples.

The field sampler is personally responsible for the care and custody of the samples collected until they are transferred or dispatched properly. The QA Project Manager determines whether proper custody procedures were followed during the field work and decides whether additional samples are required.

### **7.2.2 Transfer of Custody and Shipment**

During shipment, samples are accompanied by a Chain-of-Custody Record (Appendix A). When transferring samples, the individuals relinquishing and receiving the samples will sign, date, and note the time and condition of the samples on the record. This record documents sample custody transfer.



Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate Chain-of-Custody Record accompanying each shipment. Shipping containers will be sealed for shipment to the laboratory. The method of shipment, courier name(s), and other pertinent information are entered in the "Remarks" section of the Chain-of-Custody Record. Once received at the laboratory, laboratory custody procedures will apply.

### **7.2.3 Laboratory Custody Procedures**

A designated sample custodian accepts custody of the shipped samples and verifies that the information on the Sample Identification Label matches that on the Chain-of-Custody Records. Additional laboratory custody procedures are described in the Laboratory Quality Assurance/Quality Control Plans (Appendix C).

## **7.3 Field Notebook**

A record of sample identification numbers will be maintained in the field notebook. Additionally, the field notebook will include a record of significant events, observations, and measurements during field investigation, as well as a record of personnel present, site conditions, drilling procedures, sampling procedures, and calibration records. Field measurements recorded on standardized forms will also be maintained in the project notebook.

Field notebooks will be signed and dated by the field personnel and kept as a permanent record. The information contained in these notebooks is intended to provide sufficient data and observations to enable participants to reconstruct events that occurred during the project and to refresh the memory of the field personnel if called upon to give testimony during legal proceedings. Corrections of erroneous entries will be made by crossing a



line through the error and entering the correct information. Corrections will be initialed and dated by the person making the entry.



## 8.0 EQUIPMENT CALIBRATION, OPERATION, AND MAINTENANCE

Field equipment used to perform various measurements during Phase 2A will include an electric sounder, a steel water level tape, an EC meter for measuring the electrical conductivity of water samples, a pH meter for measuring the pH of water samples, a field thermometer, and an OVA.

Proper maintenance, calibration, and operation of each instrument will be the responsibility of the field personnel and the lead equipment technician assigned to the project. All instruments and equipment used during the studies will be maintained, calibrated, and operated according to the manufacturer's guidelines and recommendations. At a minimum, all instruments will be inspected and calibrated upon receipt from a vendor or from another office. When in the field, a copy of the manufacturer's operation and calibration recommendations will accompany the instruments. The following guidelines will apply to equipment calibration:

- .. All equipment will be calibrated prior to field use. This includes instruments used to measure water quality parameters, water levels, and well discharge as well as air monitoring devices.
- .. The probes on the EC meter and pH meter will be thoroughly rinsed with distilled water prior to each use. Also prior to each use, the pH meter will be calibrated in known pH buffer solutions. The water sample with which EC and pH are determined will not be used to fill sample containers. Manufacturer's instructions for use of each instrument will be followed.
- .. For safety purposes and for soil sample screening, an OVA will be routinely used during all drilling operations for detection of VOC emissions. When the OVA is turned off and disassembled for transport to a new work area, the new work area will be considered a measurement station. The OVA calibration will be checked at each measuring station by using a 95 ppm methane gas in air mixture.



The OVA will be considered calibrated if the reading is within  $\pm$  20 percent of the calibration gas concentration. Calibration gases of approximately 100 ppm will be used. The instrument will be sent to the manufacturer for recalibration when determined to be necessary by calibration check.

- .. Electrical conductivity and pH meters will be calibrated twice daily and after each maintenance or repair, to ensure that instrument drift or malfunction has not occurred. Thermometers will be checked against other thermometers prior to field use.
- .. Steel tapes and electric sounders used for water level and well depth measurements will be inspected regularly for kinks, stretching, or worn markings.
- .. Electric sounders with potentially moveable markers will be calibrated with a steel tape prior to each round of water level measurements.

A routine schedule and record of field instrument calibration will be maintained throughout the duration of the study. Detailed procedures for calibration, operation, and maintenance of laboratory equipment have been presented (**Appendix C**).



## 9.0 ANALYTICAL PROCEDURES

All groundwater samples collected during this project will be analyzed for the Target Chemicals outlined in Section 5.1, Page 8 by the primary laboratory, Brown & Caldwell Laboratories, Pasadena, California. At a minimum, soil and sediment samples will be analyzed for total DDT and total BHC, with additional analysis for VOC's where previous results or field screening indicate a potential for their occurrence. The laboratory report shall include volatile non-Target Chemicals if they are detected using the analytical methods proposed in this QAPP. Groundwater samples will also be analyzed for common ions to characterize the water in the Bellflower aquitard and the Gage and Lynwood aquifers. Laboratory analyses will be performed in accordance with the standard analytical procedures established by the EPA (EPA, 1986). The analytical methods, preparation methods and laboratory method detection limits (MDL) for Target Chemicals have been summarized (Table 1). Laboratory check samples will be collected from approximately 10 percent of the project monitor wells. Laboratory check samples will be submitted to Analytical Technologies, Inc. for Target Chemical analyses during each groundwater sampling round. These data will be compared with Brown & Caldwell Laboratories results as an additional check on laboratory performance. The Laboratory Quality Assurance/Quality Control Plans are included (Appendix C).



## **10.0 DATA MANAGEMENT, VALIDATION, AND REPORTING**

All raw data generated from project sampling tasks or field measurement activities which are used in preparing project reports will be appropriately identified and will be included in a separate appendix within the project reports.

### **10.1 Analytical Data**

Validation of the analytical data will be directed by the Quality Assurance Project Manager. Laboratories will submit results supported by sufficient back-up data and QA/QC results to enable the reviewer to determine the quality of the data. Validity of the data will be determined based on the data assessment procedures outlined in Section 11, Page 47 and Section 14, Page 55. Where test data have been reduced, the method of reduction will be described in the report.

Backup QA/QC data will be provided to the EPA as outlined in Appendix C-3. The backup QA/QC data will be provided in a format to be determined following discussion with the project laboratories and EPA.

### **10.2 Field Measurement Data**

Validation of data obtained from field measurements will be directed by the Quality Assurance Project Manager. Validity of the data will be determined by checking calibration procedures utilized in the field and by comparing the data to previous measurements obtained at the same site. Large variations will be examined in association with changes in local groundwater conditions and general groundwater trends. Variations in data which cannot be explained by local changes will be assigned a lower level of validity and will be used for limited purposes.





## 11.0 QUALITY CONTROL PROCEDURES

Quality control procedures are specific field and laboratory procedures used to determine accuracy, precision, and completeness of sample data. Quality control procedures for laboratory analysis and field activities, including sample collection and field measurements, are described below.

### 11.1 Laboratory Quality Control Procedures

Laboratory quality control procedures are described in Appendix C. Laboratory quality control procedures include the following:

- .. One set of calibration standards, either single point or full range, will be analyzed during each 8-hour shift.
- .. One set of reagent and solvent blanks will be analyzed daily.
- .. At least one sample will be analyzed in replicate with each batch of 10 or fewer samples.
- .. At least one spike sample will be analyzed with each batch of samples.
- .. An EPA Quality Control sample, National Bureau of Standards certified sample, or other independent sample will be analyzed with every 10 samples or one each day if fewer than 10 samples are analyzed.



## 11.2 Field Quality Control Procedures

Quality control of field data obtained during sampling and from measurement equipment will be accomplished by following the guidelines specified in Section 5, Page 7 and Section 6, Page 33, and by performing proper calibration procedures at the intervals specified in Section 8, Page 43. In addition, the QC methods discussed below shall be implemented.

### 11.2.1 Quality Control Procedures for Sample Collection

Quality control procedures to be conducted during sample collection will include the following:

- .. Duplicate samples will be taken at a rate of 10 percent per groundwater or soil sampling round or one per day minimum. Duplicate sediment samples will be collected at an overall rate of 10 percent of the total number of sediment samples. Duplicate sediment samples will be collected by selecting one of every ten consecutive samples for duplicate analyses. Duplicate samples will not be labeled as duplicates, but will be labeled in a manner similar to other samples so the laboratory will not know which samples are duplicates.
- .. Field blanks will be prepared at a rate of one per sampling day.
- .. Trip blanks for EPA Method 624 analysis will be included in each sample shipment container containing field samples for VOC analysis.
- .. Laboratory check samples will be taken at a rate of 10 percent per groundwater sampling round and delivered to Analytical Technologies, Inc. (ATI) in San Diego for Target Chemical analyses.



- .. Split samples will be provided to the EPA according to procedures outlined in Section 5.4, Pages 25 and 26.

### **11.2.2 Quality Control Procedures for Field Measurements**

#### **A. WATER LEVEL MEASUREMENTS**

As stated in Section 6.2, Page 34, water level measurements will be taken by utilizing either an electric well sounder or a graduated steel tape. Prior to obtaining measurement data, field personnel will verify that the instrument has been properly calibrated (Section 6.2, Page 34 and Section 8, Page 44).

At each location and/or time interval, a minimum of two measurements will be taken. Both measurements will be recorded in the field notebook or on the appropriate field data form. Measurements should be made to the nearest 0.01 foot.

In addition to replicate measurements, the data should be compared to previous measurements obtained at the well site. If large discrepancies exist from previous measurements which cannot be explained by local groundwater conditions, changes, or trends, the equipment operation should be checked and the measurements repeated. If possible, an alternative instrument should be utilized to verify the accuracy of the data.

#### **B. WATER QUALITY PARAMETERS**

Measurements of EC, pH, and temperature will be made during each groundwater sampling event. Prior to obtaining measurement data, field personnel will properly calibrate each instrument (Section 6.3, Page 35 and Section 8, Page 43). For EC and pH, reference solutions will be used to properly calibrate the instrument.



When obtaining data for water quality parameters, measurements should be compared with previous data and examined for variations. If variations greater than  $\pm 10$  percent exist which cannot be accounted for by changes in field conditions and/or water quality stabilization, the instrument used should be recalibrated and the measurements repeated. The most accurate measurement will be determined by the field technician and recorded in the field notebooks or on the appropriate field data form. When possible, a backup measuring device will be utilized to verify the data.

#### C. DISCHARGE MEASUREMENTS

Well discharge rates will be measured by the method outlined in Section 6.4, Page 37. Quality control will be achieved by performing periodic measurements. If the variation from the previous measurement exceeds 10 percent, the discharge measurement will be repeated.

Discharge data will be compared to measurements taken at the well site during previous sampling rounds. If variations between measurements exceeds 20 percent and cannot be accounted for by changes in pumping equipment or piping, the containers used to measure well discharge will be recalibrated.



## 12.0 PERFORMANCE AND SYSTEMS AUDITS

The Quality Assurance Manager will monitor and audit the performance of the QA procedures outlined in the QAPP. The Quality Assurance Manager will conduct field and office audits which will ensure that the information being gathered is reliable and of good quality. Audits will occur soon after the commencement of field activities. Subsequent audits will verify the effectiveness of any corrective actions implemented after previous audits.

### 12.1 Field Audits

The Quality Assurance Manager may schedule audits of field activities at various times to evaluate the execution of sample identification, sample control, chain-of-custody procedures, field documentation, instrument calibration, and field measurement and sampling operations.

Field documents pertaining to sample identification and control will be examined for completeness and accuracy. Field notebooks and field data forms will be reviewed to see that they are dated and signed and that the contents are legible, written in ink, and contain accurate and complete documentation of project activities. Because the field notebooks and field data forms provide the basis for reports written later, they will contain only facts and observations. Language will be objective, factual, and free of personal interpretations or terminology that might prove inappropriate.

The auditor will also check to see that chain-of-custody procedures are being followed and that samples are being kept in custody at all times and are sealed to prevent tampering.

Sampling operations will be evaluated to determine if they are being performed as stated in the QAPP and SAP or as directed by the Project Manager. The auditor will check to determine that the appropriate number of samples are being collected, that the samples are being placed in proper containers,



and that proper preservation, packaging, and shipment protocols are being followed.

Field measurement activities will be evaluated to determine if they are performed according to guidelines in Section 6, Page 33 and Section 8, Page 43 of this document. The auditor will spot-check various instruments for proper calibration and proper frequency of calibration, and will also check to ensure that the techniques performed using these instruments are providing accurate data.

## **12.2 Office Audits**

Once a field project has been completed, the individual files will be assembled, organized, and securely stored. The documents will be examined to determine whether all necessary items such as signatures, dates, and project codes are included and determine whether they are being handled and stored in the proper manner.

In addition to the formal audits performed, the Quality Assurance Project Manager will continually review product quality as draft documents are produced, and will ensure that the project is being performed in accordance with approved quality assurance procedures. Prior to the production of a draft report, all work products will be reviewed by senior project staff. This review will include verification of calculation briefs, test analyses, field measurements, graphs, tables, and any document which involves information generated from the field data.

## **12.3 Laboratory Audits**

Internal laboratory audits are performed periodically as part of the formal laboratory certification requirement for analyzing public drinking water systems. The laboratory audit is required to monitor the capability



and performance of the total measurement system. The audit includes a careful evaluation of equipment, standard operating procedures, and quality control procedures (Appendix C).



### 13.0 PREVENTATIVE MAINTENANCE

Preventative maintenance for quality assurance includes those tasks that must be carried out to minimize downtime of the measurement systems. Procedures for preventative maintenance during the Phase 2A field activities include the following:

- .. Instruments for field measurements will be calibrated and checked before use (Section 8, Page 43).
- .. Spare parts for instruments such as pH probes will be kept on hand in case of equipment failure.
- .. When practical, backup equipment will be available. For example, a steel water level tape will serve as backup for an electric sounder.
- .. Sufficient well construction materials will be on hand to account for monitor well variability as dictated by geologic conditions.
- .. Sampling locations and procedures will be identified and reviewed each day prior to field work.
- .. Additional materials for potential additional sample preparation such as containers, caps, and forms will be available on-site.
- .. Laboratory equipment downtime will be minimized by proper calibration and maintenance (Appendix C).





## 14.0 DATA ASSESSMENT PROCEDURES

As part of QA/QC for the RIW, routine procedures will be used to assess the precision, accuracy, and completeness of data. The results of the analysis of laboratory sample spikes will be used to determine laboratory accuracy. The results of the analysis of laboratory duplicates will be evaluated to determine laboratory precision. Data generated by field duplicate analysis will be used to evaluate the combined precision of the sampling and laboratory procedures. Data assessment procedures to evaluate accuracy, precision, and completeness are described below.

### 14.1 Accuracy

Accuracy is defined as the percent recovery from a spiked sample. A spiked sample is prepared by adding a known amount of a pure compound to the environmental sample. The compound added is the same as that being assayed for in the environmental sample. These spikes simulate the background and interferences found in the actual samples. The calculated percent recovery of the spike is evaluated as a measure of the accuracy of the total analytical method. When there is no change in volume due to the addition of the spike, accuracy is calculated as follows:

$$P = \frac{(A-B) 100}{C}$$

P = Percent Recovery

A = Concentration in spiked sample (sample and spike)

B = Concentration in sample

C = Known concentration of spike compound

Percent recovery acceptance criteria for Target Chemicals published by the EPA (1986) will be used to evaluate the overall accuracy of the analytical results. The acceptance criteria will be used as the standard for accuracy when calculating completeness.



## 14.2 Precision

Precision is defined as the relative percent difference between duplicates of the same sample. Because of the limited number of replicate samples that can be analyzed in environmental samples using gas chromatograph techniques, precision cannot be evaluated in terms of standard deviations. Consequently, outlier testing is not possible. However, the precision of an analytical method can be evaluated in a general manner from internal lab and field duplicates by calculating the relative percent difference between the duplicate sample results:

$$PD = 2 \frac{(D1-D2)}{(D1+D2)} \times 100$$

PD = Percent Difference

D1 = First Sample Value

D2 = Second Sample Value (duplicated)

The relative percent difference will be determined for Target Chemical results from internal lab and field duplicates, and from laboratory check samples. Relative percent differences between internal lab duplicate sample results will be assessed using laboratory acceptance criteria. A relative percent difference of 35 percent will be used for assessing overall precision of internal lab duplicates for soil, sediment, and groundwater matrices. Based on historical groundwater results, a percent difference value of 50 percent will be used for assessing overall precision of the field duplicate groundwater sample and laboratory check sample analytical results.

Internal lab and field duplicate samples with Target Chemical concentrations less than or equal to 10 times the detection limit will not be incorporated into the precision analysis. Small differences in concentrations in duplicate samples result in high relative percent differences when concentrations are near the detection limit. Because field soil and sediment samples are not true duplicates, but are in fact adjacent samples which historically have shown large differences in concentration, percent difference



calculations will be evaluated based on the assumption that the actual sample concentrations for these samples may be dissimilar.

If laboratory duplicate results for Target Chemicals exceed a relative percent difference of 35 percent, corrective action will be taken by the lab and if possible the sample will be reanalyzed. If the relative percent difference from field duplicate groundwater samples exceeds 50 percent, then field sampling conditions and procedures will be reviewed and the subject monitor well will be scheduled for duplicate sample collection during the next sampling round.

### 14.3 Completeness

Completeness is described as the ratio of acceptable laboratory results to the total number of analyses requested. A completeness value of less than 90 percent indicates that corrective action may be appropriate to improve the overall quality of the data. Completeness is defined as:

$$C = \left( 1 - \frac{\text{number of impaired results}}{\text{total number of requested analyses}} \right) 100$$

Criteria for defective results may include exceeded holding times, percent recoveries that fail EPA acceptance criteria, or unsatisfactory supporting data such as dates, locations, or sample identity. A completeness value less than 90 percent will necessitate a review of sampling and laboratory procedures to determine whether any changes would be appropriate to bring about improved data quality. An analysis of sample completeness will be conducted after sampling round results are returned.



#### 14.4 Assessment

Field data will be assessed by evaluating adherence to guidelines for sampling and quality control procedures outlined in **Section 5, Page 7** and **Section 11, Page 47**. Data collected by others in nearby study areas for non-RIW programs will be reviewed and considered during the investigation. Since non-RIW data have been collected by a variety of contractors and agencies using different methods and QA/QC procedures, judgments as to their quality must be made prior to incorporation into investigations.

Useful non-RIW data may include water level measurements, chemical data on water and soil, geologic subsurface descriptions such as drillers' logs, and interpretations of the hydrologic conditions of the nearby areas.

Data that are used in the investigation but were not collected by Hargis + Associates, Inc., will be evaluated against the QAPP standards. For example, a water level measurement collected during previous investigation at a particular well will be assigned a high level of confidence if the data point is accompanied by information on the type of water level measuring device, the measuring point identification, pumping status of the measured well, construction details of the well, and the general pumping status of adjacent wells. If any of these data are missing, the recorded historical water level will be assigned a lower level of confidence and may be rejected for the analysis of historical conditions.

Historical chemical data on the nature of soil or water conditions the study areas will be similarly evaluated against the standards developed in this document. Unless information is available on the method of sample collection, the analytical methods employed, and the QA/QC procedures used, the data point will be assigned a low level of confidence.

Information on subsurface conditions is often available in the form of drillers' logs. The quality of these logs varies from well to well and from driller to driller. On the basis of field experience at the Montrose site



and review of existing site-specific literature on subsurface conditions, each well log to be used in interpretative evaluations will be subject to judgment by experienced hydrogeologists.

Data collected during this investigation will be collected according to the procedures outlined in this document. If a data point appears to deviate from a historical trend, further investigations into the collection methodology and QA/QC procedures will be initiated to resolve the questionable data point. Pending the conclusion of these evaluations, the data point may either be accepted with a high or low level of confidence or rejected.



## 15.0 CORRECTIVE ACTION PROCEDURES

Should data assessment result in the detection of unacceptable data, corrective action procedures will be developed on a case-by-case basis. Such actions may include altering procedures in the field, using a different batch of sample containers, resampling or reanalyzing samples, increasing calibration and maintenance schedules of field measurement instruments, or recommending an audit of laboratory procedures. Laboratory corrective action procedures including the rationale for reanalysis of samples, are provided (**Appendix C**). Additional samples will be collected if proper QA/QC criteria cannot be met by laboratory corrective action and if the interpretation of site conditions is critically affected by the elimination of the subject sample results. The Project Manager is responsible for initiating field corrective action procedures. The Quality Assurance Manager is responsible for approving field corrective action procedures. The EPA will be notified as soon as possible when the need for corrective action arises.



## 16.0 QUALITY ASSURANCE REPORTS

The Final Remedial Investigation report will contain a separate Quality Assurance section that summarizes data quality information collected during the project. Sampling and field measurement data will be summarized by Hargis + Associates, Inc. using standard formats for sample documentation and reporting. These data, summarized along with the raw data, will be attached to all reports.



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**TABLE 1**  
**SUMMARY OF ANALYTICAL PROCEDURES**

**TARGET CHEMICALS**

<u>ANALYTES</u>	<u>EPA METHOD</u>		<u>EPA METHOD</u> <u>SOIL PREPARATION</u> <sup>(1)</sup>	<u>DETECTION LIMIT</u> <sup>(2)</sup>	
	<u>WATER</u>	<u>SOIL</u> <sup>(1)</sup>		<u>WATER (ug/l)</u>	<u>SOIL (mg/kg)</u>
DDT, DDD, DDE	608	8080	3550	0.2	0.002
BHC (alpha, beta, delta and gamma)	608	8080	3550	0.08	0.001
Chlorobenzene	624	8240	5030	1	0.13
1,2-Dichlorobenzene	624	8240	5030	1	0.13
1,4-Dichlorobenzene	624	8240	5030	1	0.13
Chloroform	624	8240	5030	1	0.13
Benzene	624	8240	5030	2	0.25
Acetone	624	8240	5030	20	2.5

**COMMON IONS**

<u>ANALYTES</u>	<u>EPA METHOD</u> <u>WATER</u>	<u>DETECTION LIMIT</u> <sup>(2)</sup> <u>WATER (mg/l)</u>
Calcium	215.1	0.1
Magnesium	242.1	0.1
Sodium	273.1	0.1
Potassium	258.1	0.05
Alkalinity	310.1	10
Chloride	325.2	1
Sulfate	375.3 or 375.4	1
Nitrate	353.2	0.4
Fluoride	340.2	0.1
Boron	200.7 or 212.3	0.05
Silica	APHA 303 C <sup>(3)</sup>	1
Total Dissolved Solids	160.1	5

(1) EPA, 1986

(2) Laboratory Method Detection Limit (MDL)

(3) American Public Health Association, 1985

ug/l = micrograms per liter

mg/kg = milligrams per liter

mg/l = milligrams per kilogram



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**TABLE 2**  
**PROPOSED SAMPLING PROGRAM**

<u>SAMPLING LOCATION/ SAMPLE TYPE</u>	<u>SAMPLE IDENTIFICATION</u>	<u>NUMBER OF BORINGS OR WELLS</u>	<u>SAMPLING METHOD</u>	<u>DEPTH OF BORING OR WELL (Ft)</u>	<u>SAMPLE DEPTHS (Ft)</u>	<u>NUMBER OF SAMPLES PER BORING OR WELL</u>	<u>TYPE OF ANALYSES</u>
Phase 1 Monitor Wells/ Groundwater	MW-1 thru MW-5	5	Bladder Pump	75	NA <sup>(1)</sup>	1 per round	Method 608 & 624
	BF-1 thru BF-4	4	Bladder Pump	125	NA	1 per round	Method 608 & 624
	G-1 thru G-3	3	Bladder Pump	165	NA	1 per round	Method 608 & 624
	LG-1	1	Bladder Pump	205	NA	1 per round	Method 608 & 624
Phase 2A Monitor Wells/ Groundwater	MW-6 thru MW-15	10	Bladder Pump	85	NA	1 per round	Method 608, 624 & Common Ions
	BF-5 thru BF-8	4	Bladder Pump	125	NA	1 per round	Method 608, 624 & Common Ions
	G-4 thru G-7	4	Bladder Pump	180	NA	1 per round	Method 608, 624 & Common Ions
	LG-2	1	Bladder Pump	215	NA	1 per round	Method 608, 624 & Common Ions
On-Site/Soils	S301 thru S305	5	Drive Sample	60	10,20,30,40, 50,60	6	Method 8080 and 8240
	S302, S305	2	Drive Sample	60	5,20,30,50	4	TOC, pH, EC, % Moisture <sup>(2)</sup>
Normandie Avenue Ditch/Soils Transect 3	T33	1	Drive Sample	21	9,12,15,18,21	5	Method 8080 <sup>(3)</sup>
	T34	1	Continuous Core	5	1,2,3,4,5	5	Method 8080 <sup>(3)</sup>
Transect 4	T42	1	Drive Sample	21	9,12,15,18,21	5	Method 8080 <sup>(3)</sup>
	T46	1	Drive Sample	60	10,15,20,25, 30,35,40,45, 50,55,60	11	Method 8080 <sup>(3)</sup>
Transect 5	T52	1	Drive Sample	60	0,2	2	Sieve <sup>(4)</sup> and Method 8080
	T52	1	Drive Sample	60	10,15,20,25, 30,35,40,45, 50,55,60	11	Method 8080 <sup>(3)</sup>
Transect 6	T64	1	Drive Sample	21	0,1	2	TOC, pH, EC, % Moisture
	T64	1	Drive Sample	21	9,12,15,18,21	5	Method 8080 <sup>(3)</sup>

Ft = Feet.

(1) NA = Not applicable.

(2) TOC, pH, EC, % Moisture = Total Organic Carbon, pH, Electrical Conductivity, and Percent Moisture.

(3) Plus EPA Method 8240 if OVA screening indicates the presence of volatile compounds.

(4) Sieve analysis to be performed on soil samples from 0.0 to 0.5 and 1.5 to 2.0 feet bls. Grain size fractions to be analyzed by EPA Method 8080.

(5) Sieve analysis to be performed on one sediment sample collected in Dominguez Channel near the confluence of the Torrance Lateral. No chemical analysis.



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TABLE 2 (continued)  
PROPOSED SAMPLING PROGRAM

<u>SAMPLING LOCATION/ SAMPLE TYPE</u>	<u>SAMPLE IDENTIFICATION</u>	<u>NUMBER OF BORINGS OR WELLS</u>	<u>SAMPLING METHOD</u>	<u>DEPTH OF BORING OR WELL (Ft)</u>	<u>SAMPLE DEPTHS (Ft)</u>	<u>NUMBER OF SAMPLES PER BORING OR WELL</u>	<u>TYPE OF ANALYSES</u>
Southern Pacific Railroad Right-of-Way/Soils SE corner of site	SP3	1	Continuous Core	5	0,5 1,2,3,4,5	2 5	TOC, pH, EC, % Moisture Method 8080
East of railroad	SP4, SP5	2	Continuous Core	5	1,2,3,4,5	5	Method 8080
Los Angeles Department of Water and Power Right-of-Way/Soils	LA15 thru LA19	5	Hand Auger	3	0,1.5,3	3	Method 8080
	LA16	1	Hand Auger	3	0,3	2	TOC, pH, EC, % Moisture
Farmer Brothers Property/Soils Transect 2	T21 thru T27	7	Drive Sample	8	0,2,4,6,8	5	Method 8080
Dominguez Channel/ Sediment Samples	SED101 thru SED106	6	Core/Hand Auger	Location, number, and depth of samples will be determined based on results from sediment thickness survey. Discrete samples will be obtained at 1-foot intervals.			Sieve <sup>(5)</sup> , Method 8080, and Percent Organic Carbon
	SED 107, SED 108	2	Core	Samples will be composited from entire cored interval.			Method 8080 and TOC
Background Sediment Sample	BGS1	1	Core/Hand Auger	10	0,2,4,6,8,10	6	Method 8080

Ft = Feet.

(1) NA = Not applicable.

(2) TOC, pH, EC, % Moisture = Total Organic Carbon, pH, Electrical Conductivity, and Percent Moisture.

(3) Plus EPA Method 8240 if OVA screening indicates the presence of volatile compounds.

(4) Sieve analysis to be performed on soil samples from 0.0 to 0.5 and 1.5 to 2.0 feet bls. Grain size fractions to be analyzed by EPA Method 8080.

(5) Sieve analysis to be performed on one sediment sample collected in Dominguez Channel near the confluence of the Torrance Lateral. No chemical analysis.

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TABLE 3

## SUMMARY OF QUALITY CONTROL FIELD SAMPLES

<u>SAMPLE TYPE</u>	<u>SAMPLE DESCRIPTION</u>		<u>COLLECTION FREQUENCY</u>	<u>TYPE OF ANALYSIS</u>
GROUNDWATER	FIELD DUPLICATE AND LABORATORY CHECK	ACTUAL SAMPLE LOCATION	FIELD DUPLICATE SAMPLE ID	
	ROUND 1	MW-6 MW-11 BF-6 G-7	MW-600 MW-1100 BF-600 G-700	Field duplicate samples collected at a rate of 10 percent of monitor wells sampled or one per day minimum.
	ROUND 2	MW-9 MW-14 BF-8 G-5	MW-900 MW-140 BF-800 G-500	
	FIELD BLANK	SAMPLE PREPARATION LOCATION	SAMPLE ID	
	ROUND 1	MW-6 WELLHEAD MW-11 WELLHEAD BF-6 WELLHEAD G-7 WELLHEAD	WB-1 WB-1 WB-1 WB-1	One per day
	ROUND 2	MW-9 WELLHEAD MW-14 WELLHEAD BF-8 WELLHEAD G-5 WELLHEAD	WB-1 WB-1 WB-1 WB-1	
	TRIP BLANK PREPARED BY BROWN & CALDWELL	SAMPLE ANALYZED BY		
	ROUND 1	BROWN & CALDWELL LAB. ATI	TB-1 TB-2	One sample per shipping container* One sample per shipping container*
	ROUND 2	BROWN & CALDWELL LAB. ATI	TB-1 TB-2	
				Method 608 and 624

\* If more than one groundwater sample per shipping container.  
ATI = Analytical Technologies, Inc.



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**TABLE 3 (continued)**  
**SUMMARY OF QUALITY CONTROL FIELD SAMPLES**

<u>SAMPLE TYPE</u>	<u>SAMPLE DESCRIPTION</u>	<u>COLLECTION FREQUENCY</u>	<u>TYPE OF ANALYSIS</u>
<b>SOIL/ON-SITE</b>	Theoretical field duplicate, sample tube adjacent to primary soil sample	10 percent or one sample per day, minimum	Method 8080 and 8240
	Equipment rinsate	One sample per day collected from one soil sampler	Method 608 and 624
<b>SOIL/OFF-SITE</b>	Theoretical field duplicate, sample tube adjacent to primary soil sample	10 percent or one sample per day, minimum	Method 8080 <sup>1</sup>
	Equipment rinsate	One sample per day collected from one soil sampler	Method 608 <sup>1</sup>
	Normandie Avenue Ditch particle size rinse water	One sample composited per soil sample	Method 608
	Normandie Avenue Ditch sieve analysis equipment rinsate	One sample composited per soil sample	Method 608
<b>SEDIMENT</b>	Theoretical field duplicate	One sample collected for every ten consecutive samples	Method 8080 and total organic carbon
	Equipment rinsate	One sample per day collected from one sediment sampler	Method 608

**Notes:**

- 1) In Normandie Avenue Ditch samples, EPA Method 8240 or 624 will be used if VOC screening results indicate the presence of volatile compounds.



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**TABLE 4**  
**SAMPLE HANDLING PROTOCOL**

<u>TYPE OF ANALYSIS</u>	<u>NUMBER, TYPE, AND SIZE OF CONTAINER</u>	<u>SAMPLE VOLUME</u>	<u>PRESERVATION</u>	<u>PRE-EXTRACTION HOLDING TIME</u>	<u>POST-EXTRACTION HOLDING TIME</u>	<u>CONTAINER CLEANING</u>
<b><u>WATER SAMPLES</u></b>						
Purgeable (Volatile) Organics (EPA Method 624)	Two 40-ml glass vials, teflon-backed septum	Vials filled completely, no air space	Wrap in plastic bag, cool to 4°C in ice chest	14 days	NA*	Bottles and septa washed with detergent, rinsed with distilled water, and dried one hour at 105°C.
Pesticides (EPA Method 608)	Two 1-liter amber glass bottles with teflon-lined caps	Bottles filled 5/6 full	Wrap in plastic bag, cool to 4°C in ice chest	7 days	40 days	Bottles and cap liners rinsed with methylene chloride and dried by vacuum or other safe means until no solvent remains.
Common Ions	One 1-liter poly- ethylene bottle	Bottle filled completely	Wrap in plastic bag, cool to 4°C in ice chest	14 days	NA*	Container washed with detergent and rinsed with distilled water.
Nitrate	One 100-ml poly- ethylene bottle	Bottle filled completely	Preserve sample with sulfuric acid to pH of less than 2; wrap in plastic bag, cool to 4°C in ice chest	28 days	NA*	Container washed with detergent and rinsed with distilled water, pretreated with sulfuric acid.

\* NA = Not applicable



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TABLE 4 (continued)  
SAMPLE HANDLING PROTOCOL

<u>TYPE OF ANALYSIS</u>	<u>NUMBER, TYPE, AND SIZE OF CONTAINER</u>	<u>SAMPLE VOLUME</u>	<u>PRESERVATION</u>	<u>PRE-EXTRACTION HOLDING TIME</u>	<u>POST-EXTRACTION HOLDING TIME</u>	<u>CONTAINER CLEANING</u>
<u>SOIL SAMPLES</u>						
Volatile Organics (EPA Method 8240)	One sealed brass tube sleeve with teflon- lined film and plastic cap	6" x 2" tube. 2 gram aliquot required	Wrap in plastic bag, cool to 4°C in ice chest	**	**	Sampler washed with detergent; rinsed with tap water, methanol, distilled water.
Pesticides (EPA Method 8080)	One sealed brass tube sleeve with teflon- lined film and plastic cap	6" x 2" tube. 10 gram aliquot required	Wrap in plastic bag, cool to 4°C in ice chest	7 days	30 days	Sampler washed with detergent; rinsed with tap water, methanol, distilled water.
EC, pH, Percent Moisture, & Total Organic Carbon	One sealed brass tube sleeve with teflon- lined film and plastic cap	6" x 2" tube.	Wrap in plastic bag, cool to 4°C in ice chest	***	***	Sampler washed with detergent; rinsed with tap water, methanol, distilled water.
<u>SEDIMENT SAMPLES</u>						
Pesticides (EPA Method 8080)	One sealed brass tube sleeve with teflon- lined film and plastic cap	6" x 2" tube. 10-gram aliquot required	Wrap in plastic bag, cool to 4°C in ice chest	7 days	30 days	Sampler washed with detergent; rinsed with tap water, methanol, distilled water.
	or: Two 1/2-liter wide- mouth glass jars, teflon-lined threaded caps	Jars filled completely 10-gram aliquot required	Wrap in plastic bag, cool to 4°C in ice chest			Jars and cap liners rinsed with methylene chloride and dried by vacuum or other safe means until no solvent remains.
Total Organic Carbon	One sealed brass tube sleeve with teflon- lined film and plastic cap	6" x 2" tube.	Wrap in plastic bag, cool to 4°C in ice chest	***	***	Sampler washed with detergent; rinsed with tap water, methanol, distilled water.
	or: Two 1/2-liter wide- mouth glass jars, teflon-lined threaded caps	Jars filled completely	Wrap in plastic bag, cool to 4°C in ice chest			Jars and cap liners rinsed with methylene chloride and dried by vacuum or other safe means until no solvent remains.

\*\* Extraction and analysis will be conducted within 14 days of sample collection.

\*\*\* Extraction holding time not established at this writing.



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TABLE 5

**VOLUMETRIC ESTIMATES AND PROPOSED HANDLING  
OF INVESTIGATION-DERIVED MATERIALS  
FOR PHASE 2A REMEDIAL INVESTIGATION**

<u>DISPOSABLE MATERIAL</u>	<u>ESTIMATED VOLUME (Gallons)</u>	<u>TEMPORARY STORAGE</u>	<u>FINAL DISPOSAL OPTIONS</u>
<b>Water</b>			
Decontamination water	15,000	Baker Tank	On-site evaporation; On-site treatment and subsequent discharge to storm sewer; or Off-site water treatment facility
Groundwater	36,000	Baker Tank	On-site evaporation; On-site treatment and subsequent discharge to storm sewer; or Off-site water treatment facility
<b>Rotary Drilling Materials</b>			
Drilling fluids	80,000	Baker Tank	Landfill or site remediation
Drill cuttings	30,000	Roll-off bins and 55-gallon drums	Landfill or site remediation
<b>Auger Cuttings</b>			
Soil borings	10,000	Roll-off bins and 55-gallon drums	Site remediation or landfill
Shallow monitor wells	8,000	Roll-off bins and 55-gallon drums	Landfill
Solid Waste	4,000	Roll-off bins and 55-gallon drums	Landfill



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**TABLE 6**  
**SUMMARY OF EXPOSURE LIMITS<sup>(1)</sup>**

<u>TARGET CHEMICAL</u>	<u>TLV<sup>(2)</sup></u> <u>(ppm)</u>	<u>PEL<sup>(3)</sup></u> <u>(ppm)</u>
DDT (Dichloro-diphenyl-trichloroethane)	1	1
BHC (Benzenehexachloride/Lindane)	0.5	0.5
Chlorobenzene	75	75
Benzene	10	10
Chloroform	10	50

- (1) See Health and Safety Plan, Table 2, for a detailed list of primary hazards and exposure limits.
- (2) Threshold Limit Value - Airborne concentrations of a substance that is believed to present no adverse effects to nearly all workers, based on 8 hours per day, 5 days per week exposure over 40 to 50 years. These recommended values are set by American Conference of Governmental Industrial Hygienists.
- (3) Permissible Exposure Limit - A standard similar to the TLV that is a legal limit set by the Occupational Safety and Health Administration (OSHA).
- ppm = parts per million.



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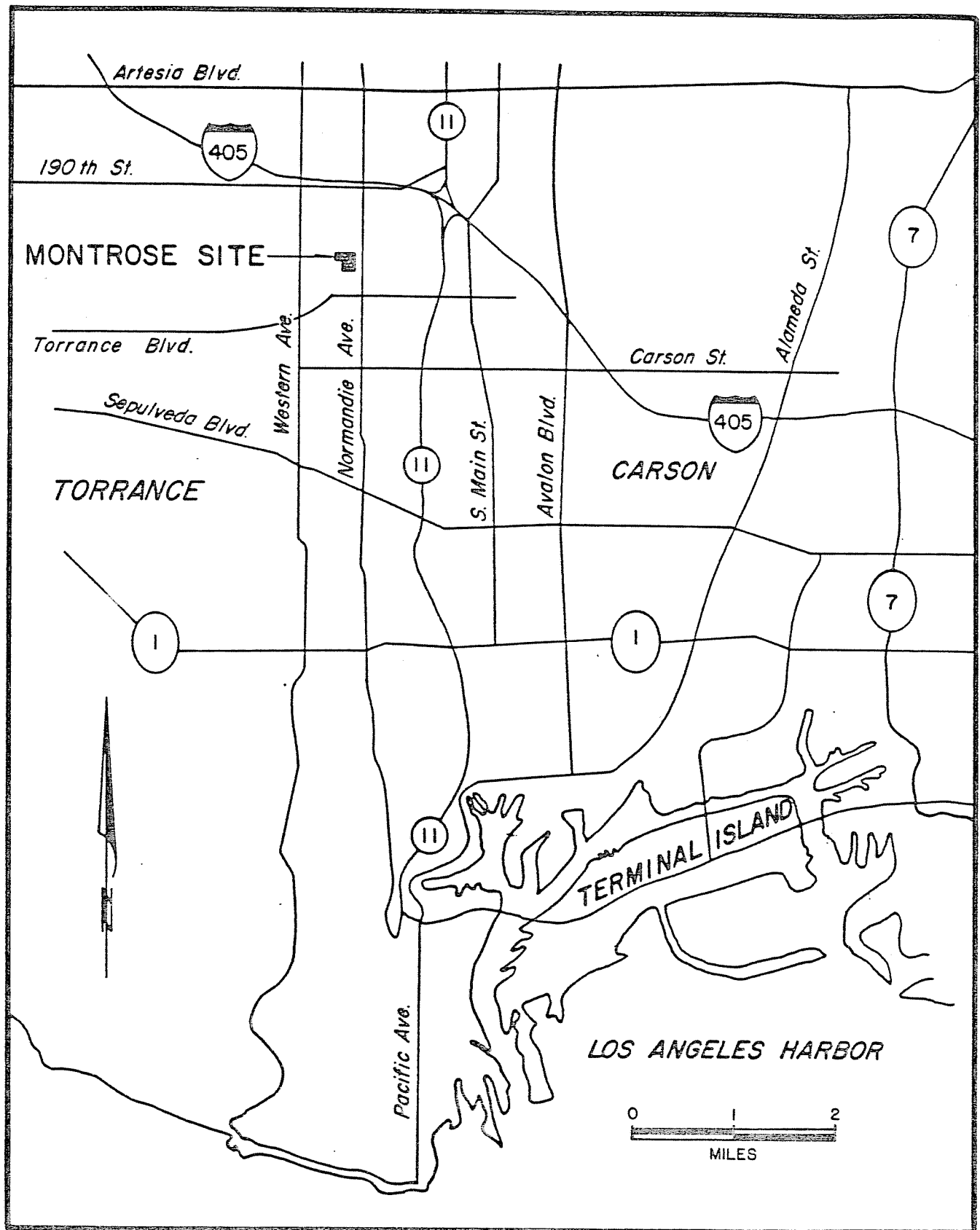
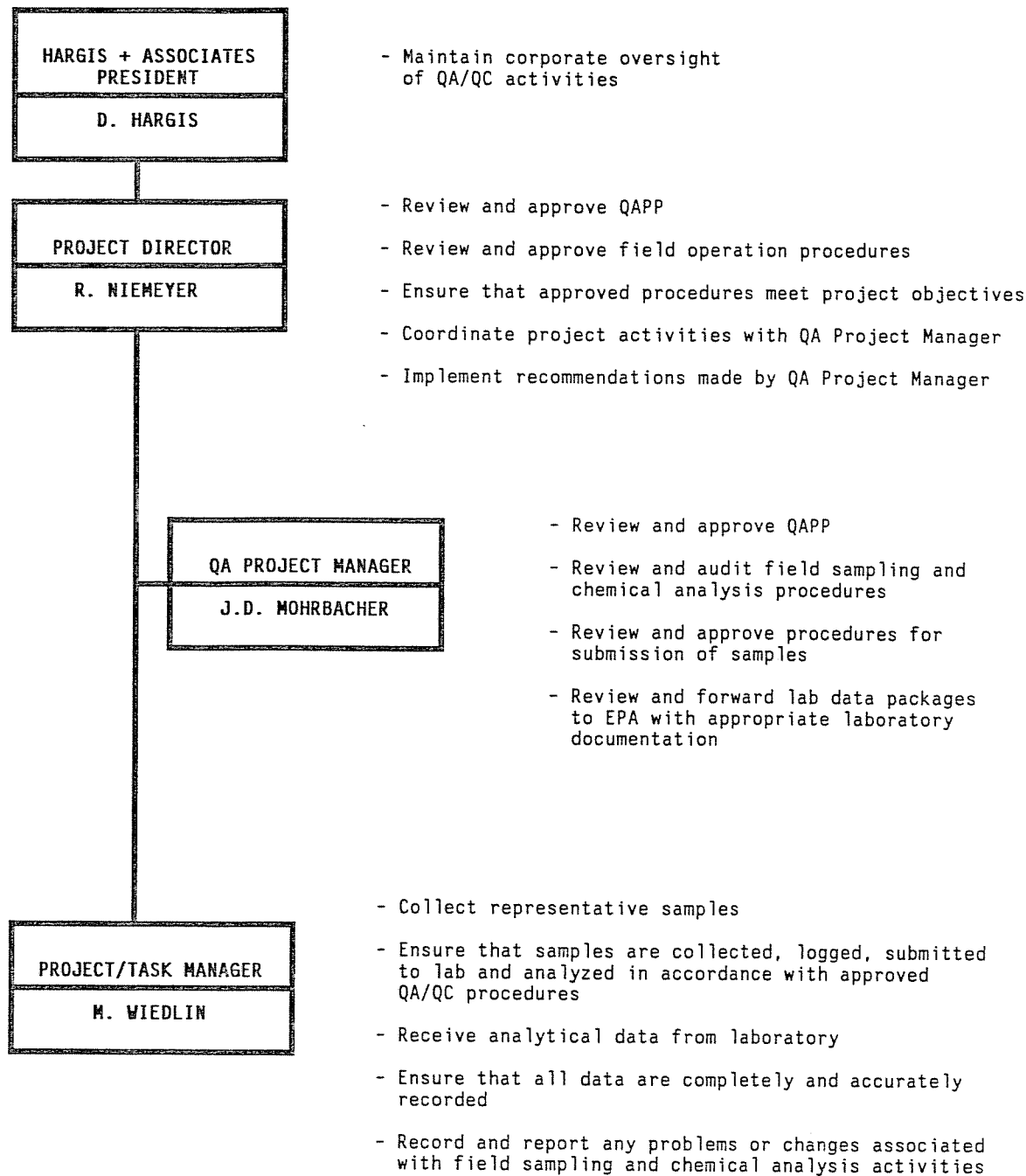


FIGURE I. LOCATION OF MONTROSE SITE



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**FIGURE 2. QUALITY ASSURANCE ORGANIZATIONAL CHART**



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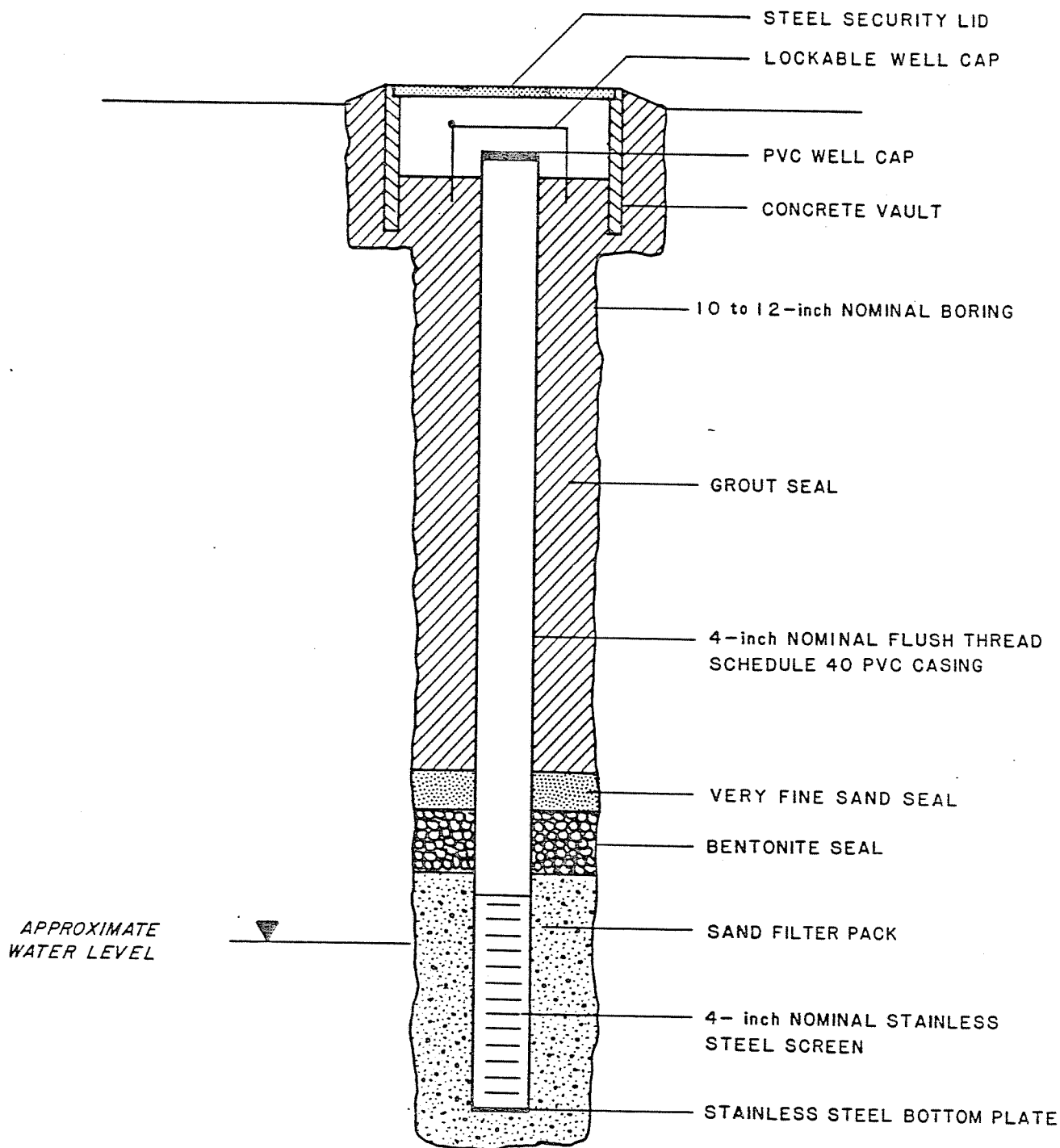


FIGURE 3. GENERALIZED CONSTRUCTION DIAGRAM FOR HOLLOW  
STEM AUGER MONITOR WELL COMPLETIONS



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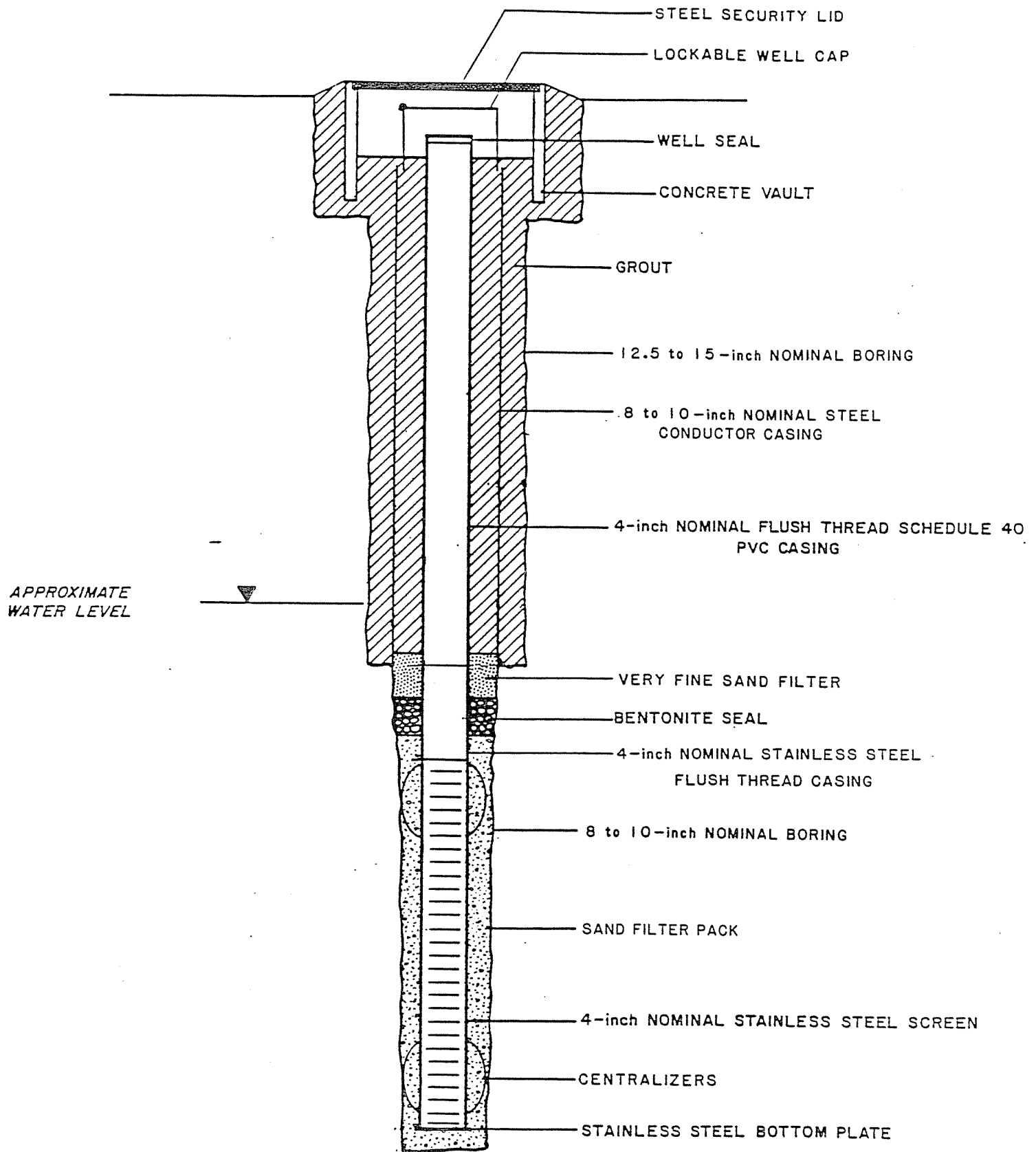


FIGURE 4. GENERALIZED CONSTRUCTION DIAGRAM FOR FLUID  
ROTARY MONITOR WELL COMPLETIONS



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## APPENDIX A

### SAMPLE CONTROL/CHAIN-OF-CUSTODY DOCUMENTS



**APPENDIX A**

**SAMPLE CONTROL/CHAIN-OF-CUSTODY DOCUMENTS**

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**Figure**

<b>A-1</b>	<b>SAMPLE IDENTIFICATION LABEL</b>
<b>A-2</b>	<b>CHAIN-OF-CUSTODY RECORD/LABORATORY REQUEST SCHEDULE</b>
<b>A-3</b>	<b>TRANSMITTAL LETTER</b>



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Consultants in Hydrogeology

2223 Avenida De La Playa, Suite 300  
La Jolla, California 92037 (619) 454-0165

Client		Date
Project #	Sample ID	
Initials	Time	
Analyze for		
Preservative/Special Instructions:		

FIGURE A-1. SAMPLE IDENTIFICATION LABEL



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PROJECT NAME					PROJECT NUMBER					ANALYSES REQUIRED										REMARKS, PRESERVATIVES, SPECIAL HANDLING
PROJECT MANAGER					TYPE & NUMBER OF CONTAINERS					CONTAINER LOT NUMBER										
SAMPLERS					6"x2" BRASS TUBE 40ml VOA VIAL 1-lit AMBER GLASS 16 oz GLASS JAR					MONOCHLOROBENZENE EPA 8240/624 DICHLOROBENZENE EPA 8240/624 BENZENE EPA 8240/624 CHLOROFORM EPA 8240/624 ACETONE EPA 8240/624 TOTAL DDT w/ISOMERS EPA 8080 TOTAL BHC w/ALL ISOMERS EPA 8080/608										
SAMPLE NUMBER		DATE	TIME	SAMPLE TYPE																
LOCATION CODE	SEQUENCE/DEPTH	SAMPLED	SAMPLED	COMPOSITE OR GRAB	SOIL OR WATER															
-	/	/86																		PRESERVED ON ICE
-	/	/86																		PRESERVED ON ICE
-	/	/86																		PRESERVED ON ICE
-	/	/86																		PRESERVED ON ICE
-	/	/86																		PRESERVED ON ICE
-	/	/86																		PRESERVED ON ICE
-	/	/86																		PRESERVED ON ICE
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-	/	/86																		PRESERVED ON ICE
-	/	/86																		PRESERVED ON ICE
-	/	/86																		PRESERVED ON ICE
-	/	/86																		PRESERVED ON ICE

SIGNATURE		COMPANY/SHIPPING METHOD	DATE	TIME	REMARKS/CONDITION OF SAMPLES
RELINQUISHED BY:		HARGIS + ASSOCIATES			
RECEIVED BY:					
RELINQUISHED BY:					
RECEIVED BY:					
RELINQUISHED BY:					
RECEIVED BY:					

SHIP TO: BROWN & CALDWELL  
373 S. Fair Oaks Ave.  
Pasadena, CA 91105  
Attn: Bob Peak

SEND RESULTS TO: HARGIS + ASSOCIATES San Diego  
Attn. Roger Niemeyer

SEND INVOICE TO: HARGIS + ASSOCIATES San Diego  
Attn. Roger Niemeyer



HARGIS + ASSOCIATES, INC.



**HARGIS + ASSOCIATES, INC.**  
Consultants in Hydrogeology

2223 Avenida De La Playa Suite 300  
La Jolla, California 92037  
(619) 454-0165

Job No.: \_\_\_\_\_

Date: \_\_\_\_\_

Brown & Caldwell Laboratories  
373 S. Fair Oaks Avenue  
Pasadena, CA 91105

Please call upon arrival

ATTENTION: BOB PEAK

Transmitted herewith are \_\_\_\_\_ parcels containing \_\_\_\_\_ soil/water samples collected from \_\_\_\_\_ sampling stations. The samples were shipped via \_\_\_\_\_ on the above date. The contents of the sample shipment are shown on the attached chain-of-custody record.

The laboratory representative who accepts custody of these samples from the shipping company should, upon receipt, record the number and condition of the samples and acknowledge receipt of the samples by signing and returning the custody record.

Please perform the analyses outlined on the attached chain-of-custody and include all data on sample labels in the final laboratory report. The date each sample was received by the laboratory and the dates of extraction, analysis, etc., should also appear on the laboratory report. Please report all analytical results to \_\_\_\_\_ of the H+A office in \_\_\_\_\_.

Payment is contingent upon adherence to method requirements for sample handling, preservation, preparation, and analysis, or as otherwise agreed to by the Hargis + Associates, Inc. Project Manager before work commences.

If you have any questions concerning the requested analyses or the integrity of the sample(s) in question, please contact the above project manager, me, or our lab coordinator.

Results of your analyses should be reported to our office as soon as possible.

Sincerely,  
HARGIS + ASSOCIATES, INC.

**FIGURE A-3. TRANSMITTAL LETTER**

Phoenix office:  
2222 South Dobson Road Suite 401  
Mesa, Arizona 85202  
(602) 345-0888

Tucson office:  
1735 East Fort Lowell Road Suite 5  
Tucson, Arizona 85719  
(602) 881-7300





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## APPENDIX B

### FIELD MEASUREMENT FORMS



**APPENDIX B**

**FIELD MEASUREMENT FORMS**

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**Table**

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<b>B-2</b>	<b>INSTRUMENT CALIBRATION LOG FOR MONITOR WELL SAMPLING</b>
<b>B-3</b>	<b>STATIC WATER LEVEL DATA SHEET</b>
<b>B-4</b>	<b>MONITOR WELL SAMPLING INFORMATION</b>
<b>B-5</b>	<b>MONITOR WELL FIELD PARAMETERS</b>
<b>B-6</b>	<b>FIELD DUPLICATE, TRIP BLANK, AND FIELD BLANK LOG SHEET</b>



# LITHOLOGIC LOG

[illegible][illegible]

**TABLE B-2**  
**INSTRUMENT CALIBRATION LOG FOR GROUNDWATER SAMPLING**

**EC METER CALIBRATIONS**

DATE	TIME	EC STANDARD SOLUTION UMHOS/CM	TEMPERATURE OF SOLUTION °C	EC METER READING UMHOS/CM	METER TYPE	COMMENTS	INITIALS

**pH METER CALIBRATION**

DATE	TIME	pH BUFFER (UNITS)	TEMPERATURE OF BUFFER	pH READING (UNITS)	METER TYPE	COMMENTS	INITIALS



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**TABLE B-3**  
**STATIC WATER LEVEL DATA SHEET**

[illegible]

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## MONITOR WELL SAMPLING INFORMATION

[illegible]

.....DISCHARGE RATE.....

[illegible]

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TABLE B-6

## FIELD DUPLICATE, TRIP BLANK, AND FIELD BLANK LOG SHEET

## FIELD DUPLICATE SAMPLES

SAMPLE DATE	ACTUAL SAMPLE LOCATION	ACTUAL SAMPLE TIME	FICTITIOUS SAMPLE ID	FICTITIOUS SAMPLE TIME	ANALYTICAL METHOD	COMMENTS	INITIALS

## TRIP BLANKS

SAMPLE DATE	SAMPLE ID	FICTITIOUS SAMPLE TIME	LABORATORY PREPARATION DATE	ANALYTICAL METHOD	COMMENTS	INITIALS

## FIELD BLANKS

SAMPLE DATE	SAMPLE ID	SAMPLE TIME	PREPARATION LOCATION	BLANK WATER SOURCE AND DATE	COMMENTS	INITIALS



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## APPENDIX C

### LABORATORY QUALITY CONTROL





**APPENDIX C**  
**LABORATORY QUALITY CONTROL**

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<b>C-2</b>	<b>LABORATORY QUALITY ASSURANCE/QUALITY CONTROL PLAN, ANALYTICAL TECHNOLOGIES, INC.</b>
<b>C-3</b>	<b>LABORATORY QUALITY ASSURANCE/QUALITY CONTROL DATA PACKAGE OUTLINE</b>



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**APPENDIX C-1**

**QUALITY ASSURANCE MANUAL  
BROWN AND CALDWELL LABORATORIES**

## QUALITY ASSURANCE MANUAL

### BROWN AND CALDWELL LABORATORIES

Steven A. Fisher	President
James Hatfield	Quality Assurance Director
Daniel A. McLean	Vice President Laboratory Director, Emeryville
Edward Wilson	Vice President Laboratory Director, Pasadena
James C. Hein	Technical Director, Emeryville
Larry L. Schaleger, Ph.D.	Technical Director, Pasadena

Fifth Edition  
April, 1987

## STATEMENT OF PURPOSE

Brown and Caldwell Laboratories' Quality Assurance Program is designed to ensure the accuracy and precision of all analytical results. Our philosophy is to provide our clients with service of the highest quality available. Control of analytical results is maintained by adherence to specified operating procedures, use of quality control samples and standards, and observance of sample custody requirements. The purpose of this manual is to describe the basic elements of our overall Quality Assurance program.

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SECTION I  
GENERAL DESCRIPTION

A. Services

Brown and Caldwell Laboratories has been providing laboratory services to industry and government for 40 years. Laboratories located at Emeryville in the San Francisco Bay Area and at Pasadena in Greater Los Angeles perform a full range of analytical tests, including the following:

- Compliance monitoring for Resource Conservation and Recovery Act, National Pollution Discharge Elimination System, Title 22, and Air Resource Board
- Potable water testing
- Water and wastewater characterization
- Toxic and hazardous waste investigation
- Treatability studies
- Analytical consultation and methods development
- Priority pollutant analysis
- Agricultural pesticide analysis
- Quality control services
- Bacteriology and microbiology
- Toxicity bioassays
- Sample collection and on-site analysis
- Testing of landfills to meet Calderon Bill requirements

B. Personnel

The Division is made up of about 100 experienced chemists, biologists, and technical assistants. Half have degrees in chemistry and the remainder have other technical degrees. Several staff members hold advanced degrees in their primary field.

In addition to academic preparation, a formal program of training is carried out for new employees or those who change job assignments. A training outline is prepared by

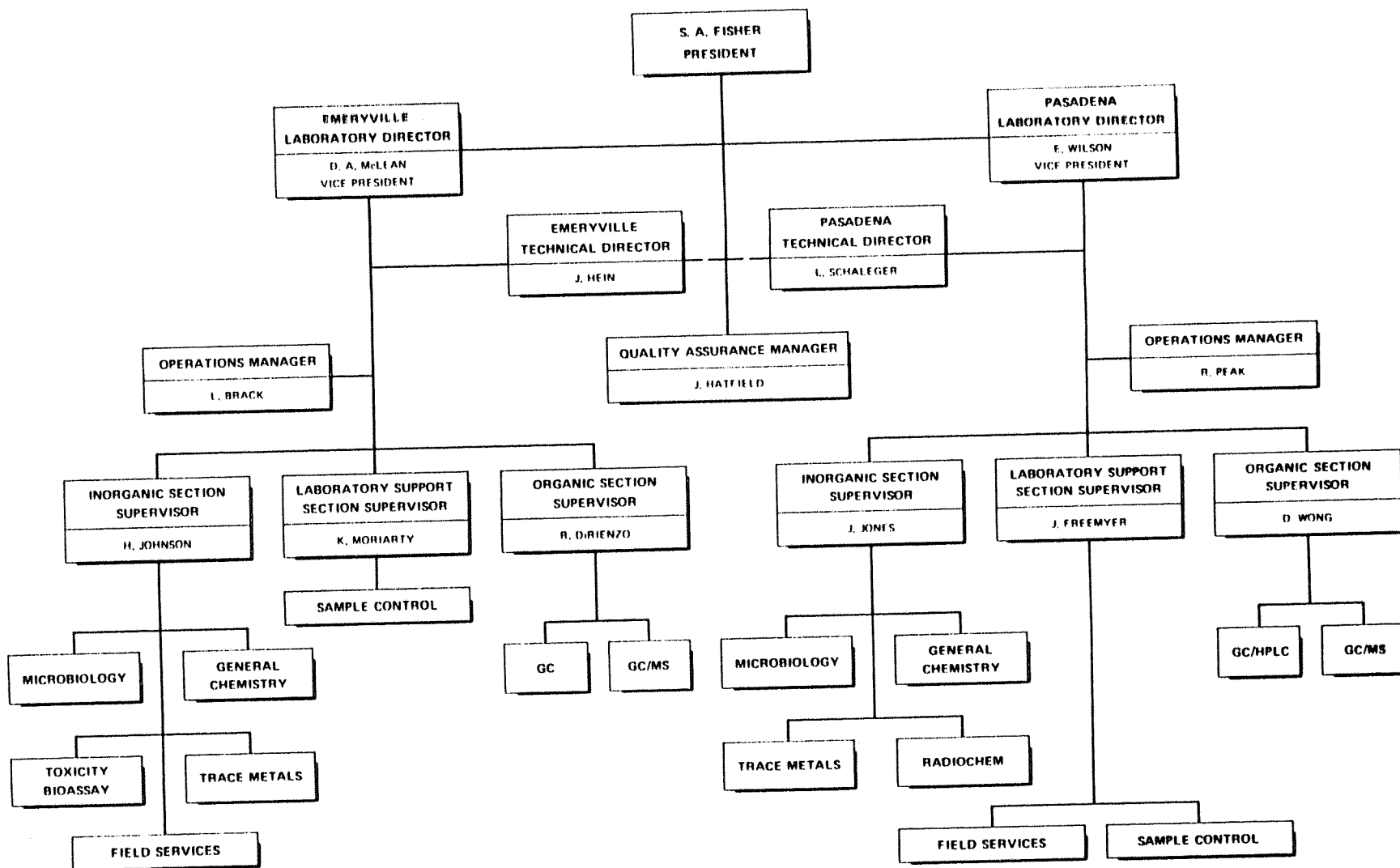
the supervisor, approved by the Technical Director, and discussed with the employee and any senior analysts who may participate in the training. At the end of three months, a technical review is carried out. Questions are asked the staff member, while being observed by a review board usually consisting of the Technical Director, the Quality Assurance Director, and the Laboratory Director or Operations Manager. In addition to the oral review, there is a practical examination of procedure. The practical exam is also followed by successful analysis of a performance check sample. This ensures that the analyst has a practical understanding of procedure and application as well as the theoretical background.

Brown and Caldwell also provides a system for professional development away from the job. Laboratory specialists are frequently sent to training sessions and seminars presented by instrument manufacturers and professional societies. Dues and tuition reimbursement are available to all employees to maintain and enhance their professional competence. Seminars are regularly presented at each laboratory by division staff members and experts from other organizations.

#### C. Structure

The staff within each laboratory is organized into three primary sections: Inorganic, Organic and Laboratory Support (Figure 1).

Each laboratory has a Director responsible for ultimate approval of all analytical work. The laboratory-wide quality assurance program is headed by a Quality Assurance Director responsible for monitoring the performance of the laboratories and for taking corrective action as needed. The Quality Assurance Director reports directly to the President of Brown and Caldwell Laboratories, and is assisted by a Technical Director in each laboratory, whose responsibilities include the resolution of technical problems and the documentation of new methods. To consistently manage workload requirements and staff resources, an Operations Manager in each laboratory reviews data production and report timeliness on a daily basis.



BROWN AND CALDWELL LABORATORIES STAFF ORGANIZATION CHART



## SECTION II

### SAMPLE HANDLING

#### A. Collection

Sampling equipment, appropriate containers and preservatives, and careful monitoring of holding times are a few of the points which must be considered to minimize possibilities for contamination or other threats to the integrity of the sample. Quality assurance begins with experienced field personnel. Sample bottles are clearly marked and all pertinent observations recorded along with the sample description, time and date of collection, and initials of the collector.

To assure proper container selection and appropriate application of preservatives, field personnel and sample control technicians are provided with EPA container and preservation guidelines. The rules they follow are contained in Table II of 40 CFR Part 136, Federal Register, October 26, 1984. Special containers, such as vials for volatile organics and amber glass bottles pretreated for organic priority pollutant analysis, are purchased from suppliers who provide certificates of compliance with EPA regulations.

#### B. Control and Custody

Chain-of-custody procedures have been established to document the identity of a sample and its handling from the time of collection until its ultimate disposal. The sampling technician in the field initiates a chain-of-custody record (Figure 2) which is provided with the bottles and remains with the sample throughout its handling. This includes the transfer of samples from the field crew to the laboratory. Custody seals are also available when required to demonstrate that a sample has been protected from tampering.

II-1

Each sample is assigned a discrete log number which, in addition to being attached to the sample container, is entered on the custody record, in the legally required sample log book, and onto the computerized data handling system. Besides the log number, the computerized record also contains the client name, sample description, sample matrix type, required analyses, and the report due date. Supplementary information such as special handling requirements may be entered as well.

Verification of sample integrity is one of the main responsibilities of the Sample Control Group. The sample is inspected to see that:

1. The sample is clearly marked and dated.
2. The sample was collected in an appropriate container.
3. The sample is properly preserved.
4. There is sufficient volume to do all the analyses required.
5. The sample is received in good condition and the custody seal is intact (if used).
6. Chain-of-custody forms match the number and description of samples.

If the above conditions are met, the sample will be assigned a log number and the relevant information is recorded. If aliquots or subsamples are to be split out, care is taken to ensure that the subsamples are representative of the original. Blending or grinding may be required.

The Sample Control Group distributes the sample (or fractions if the sample requires subdivision) into designated storage areas. Most samples are stored under refrigeration at 4 degrees C. Refrigerators are marked with test categories for convenient retrieval of samples. Volatile organic vials are segregated from other samples to prevent vapor-phase cross contamination.

## BC 1 Number-

FIGURE 2

☐ 1255 Powell Street, Emeryville, CA 94608 (415) 428-2300  
☐ 373 South Fair Oaks Avenue, Pasadena, CA 91105 (213) 681-4655

Hazardous samples will be returned to client or disposed of at client expense.

### SECTION III

#### METHODS

##### A. Sources

Brown and Caldwell makes extensive use of methods prescribed by the USEPA. Other methods are taken from Standard Methods for the Examination of Water and Wastewater, 16th Edition, APHA-AWWA-WPCF, 1985.

Primary USEPA sources of methods for the analysis of aqueous samples include:

Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA-600/4-82-057, July 1982.

Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised, March 1983.

Federal Register, 40 CFR Part 136, October 26, 1984.

Soil and other solid samples are analyzed according to the procedures of Test Methods for Evaluating Solid Waste, USEPA SW-846, July 1982.

A number of additional EPA methods are summarized in the "AB 1803 Methods Manual" issued by the California Department of Health Services, 1984.

Air analysis is based on the NIOSH Manual of Analytical Methods, 2nd and 3rd editions, issued by the National Institute of Occupational Safety and Health of the Public Health Service.

Additional methods for various sample types are taken from such sources as the United States Geological Survey (USGS), the American Society for Testing and Materials (ASTM), and the Association of Official Analytical Chemists (AOAC).

III-1

Finally, a number of special methods have been developed by Brown and Caldwell or adapted from literature sources for specialized applications. These methods are written up and maintained in a Brown and Caldwell methods reference manual.

#### B. Examples

A listing of methods used by Brown and Caldwell for priority pollutant analysis in aqueous samples and solid wastes is given in Table 1. Also included are methods for hazardous waste analysis.

TABLE 1

#### METHODS FOR PRIORITY POLLUTANT AND HAZARDOUS WASTE ANALYSIS

<u>Determination</u>	<u>EPA Method Number</u>	
	<u>Water</u>	<u>Soil, Waste</u>
Volatile organics by GC/MS	624	8240
Semivolatile organics by GC/MS	625	8270
Organochlorine pesticides and PCB by GC/ECD	608	8080
Cyanide	335.3	9010
Phenolics, total	420.1	9065
Antimony	204.2	7041
Arsenic	206.2	7060
Beryllium	210.2	7091
Cadmium	213.2	7131
Chromium	218.2	7191
Hexavalent Chromium	218.5	7196
Copper	220.2	7211
Lead	239.2	7421
Mercury	245.1	7471
Nickel	249.2	7521
Selenium	270.3	7740
Silver	272.2	7761
Thallium	279.2	7841
Zinc	289.2	7950
Ignitability	----	1010
EP Toxicity	----	1310
Chlorophenoxy Herbicides	8150	8150

Note: GC/MS is Gas Chromatography/Mass Spectrometry and GC/ECD is Gas Chromatography with Electron Capture Detection.

SECTION IV  
FACILITIES AND EQUIPMENT

A. Physical Conditions

The two Brown and Caldwell laboratories contain more than 25,000 square feet of work space. Both are equipped with refrigerated storage areas, fume hoods, central deionized water systems, and general utilities appropriate for modern analytical work. Common equipment such as ovens, incubators, and distillation apparatus are supplemented by specialized instruments described below.

B. Instruments

1. Laboratory Computer System

The multiuser information system is designed for sample and data tracking as well as automatic reporting and invoicing. The two laboratories operate from a shared data base. Hardware includes:

- a. Two ADDS Mentor computers.
- b. Two Micom 8000 twelve-channel multiplexer modems.
- c. Thirty WYSE video display terminals.
- d. Four high-speed form printers and three letter-quality printers.

2. Gas Chromatograph/Mass Spectrometers

The five GC/MS instruments include:

- a. Two Finnigan 4000 series GC/MS systems interfaced with Nova 4X and 4C computers utilizing Winchester 70 megabyte and 35 megabyte disk drives, respectively for data acquisition. Computer 4X is equipped with Finnigan 6.1 REV Superincos software while computer 4C has 5.1 REV. Cipher streamer tape cartridges are used for long-term data storage.

- b. One Finnigan OWA/1050 GC/MS interfaced with a Nova 4C computer utilizing a 70 megabyte Winchester disk drive for data collection. This data system also employs 5.5 REV Superincos Finnigan software. A 1/4 inch cartridge tape drive is used for long term data storage. The GC/MS instrument can be configured in capillary or packed column modes. A Tekmar automatic liquid sampler is also used for volatile analysis.
- c. One Finnigan Model 5100 GC/MS interfaced with a data general Nova 4X computer utilizing a 20 megabyte Winchester disk drive and a 1/4 inch cartridge cipher tape drive for longer data storage. The 5100 is equipped with a Model 8600 series autosampler.
- d. One Hewlett-Packard Model 5890/5988A GC/MS interfaced with an HP 1000 computer data system consisting of 1 megabyte main RAM memory, 132 megabyte fixed Winchester disk drive, and a 1/4 inch streamer tape for data archiving. An HP Model 7673A autosampler allows automatic sample introduction. RTE-6/UM GC/MS quantitation software is combined with the Aquarius Revision E CLP forms generation software. The system also includes HP 2934 and HP 2563 printers, two HP 150 PC "smart terminals" and Advance Link data communications software.

### 3. Gas Chromatographs

Selective detector gas chromatography is employed to solve a variety of analytical problems in which components of a general chemical class are to be distinguished from background materials not having the class-specific properties. The 20 gas chromatographs currently in use are equipped with a wide variety of detectors, including the following:

- a. Flame ionization detector. Nonselective, used for fuel fingerprinting and odor pattern matching.
- b. Thermal conductivity detector. Nonselective, used for the analysis of methane, carbon dioxide, and other gases.

- c. Electron capture detector. Moderately selective for electron capturing components such as organochlorine pesticides, PCB, and phthalates.
- d. Flame Photometric Detector. Selective for phosphorus or sulfur containing organics such as organophosphorus pesticides or sulfur gases.
- e. Hall detector. When operated in the halogen mode, highly selective for compounds such as trihalomethanes and chlorinated solvents. It can also be operated in sulfur-selective mode.
- f. Photoionization detector. Selective for photoionizable components such as aromatic solvents, esters and unsaturates.
- g. Thermionic selective detector. Selective for nitrogen and phosphorus containing organics such as organophosphorus pesticides and amines.

#### 4. Liquid Chromatograph

Altex model CS4400 liquid chromatograph (LC) is equipped with a microcomputer-controlled solvent gradient system, a multichannel chromatography data system, a variable wavelength UV/VIS detector, and heads for both analytical and preparative scale work.

#### 5. Inductively Coupled Plasma Spectrometer

The Perkin-Elmer Plasma II Inductively coupled plasma spectrometer (ICP) is equipped with a Perkin-Elmer Model 7500 Professional Computer with color graphics for full automation of sample processing and data handling. Supported with a PR 210 dot-matrix color printer, the instrument is capable of determining up to 15 different trace metals in a single analysis.

#### 6. Atomic Absorption Spectrophotometers

The six instruments in current use include Perkin-Elmer models 5000 (2), 2380 (2), 460, and 503 which are equipped to perform flame, graphite furnace, gaseous hydride, and cold vapor analyses.



7. Ion Chromatographs

A Dionex Model 10 and a Dionex Model QIC are equipped with conductivity and electrochemical detectors. They are employed for multicomponent analysis of anions and cations in complex matrices.

8. Autoanalyzers

An Alpkem Model RFA 300, a Technicon Model II, and a Lachat Model 4 are autoanalyzers routinely used for nutrient analyses. Manifolds available include NO<sub>2</sub>, NO<sub>3</sub>, PO<sub>4</sub>, Cl, SO<sub>4</sub>, CN, NH<sub>3</sub>, Cr VI, and TKN.

9. Spectrophotometers

The nine spectrophotometers available for routine use include Bausch and Lomb fixed wavelength models, Perkin-Elmer models UV/VIS 283 and 137B IR, and a Coleman model 21 flame photometer.

10. Total Organic Carbon Analyzers

Dohrmann model DC80, Beckman models 915B, and Oceanographic International Model 700 are available. Instrument selection depends on matrix type and concentration.

11. Total Organic Halide (TOX) Analyzers

Xertex (Dohrmann) model DX20 and Oceanographic Instruments model 610 TOX analyzers are available for RCRA groundwater compliance monitoring.

12. Radiochemical Counter

Gross alpha and /or beta concentrations can be determined with an Eberline model MS-3 high voltage supply and an FC-2 internal proportional counter.

13. Treatability Equipment

The Pasadena laboratory has a 700 square foot area designated for treatability studies. We have a broad array of laboratory scale equipment for evaluating the feasibility of various treatment processes. Equipment available includes biological

reactors for activated sludge treatment; ozone generator with column contactors for oxidation treatment; various carbon, mixed-media, sand, and ion exchange columns for filtration/adsorption treatments; and paddle stirrers for flocculation treatment.

#### 14. Bioassay Equipment

Bioassays are conducted in a 200 square foot temperature-controlled environmental chamber. One 350 gallon aquarium provides large scale holding capacity for acclimation of test species.

Routine equipment maintenance and instrument service is performed on an appropriate service schedule basis. Major instruments such as balances, gas chromatographs, atomic absorption spectrophotometers, and the GC/MS systems are maintained under commercial service contracts or by in-house service technicians. Calibration, sensitivity, and response checks on a daily basis establish unscheduled service needs for analytical instruments.

#### C. Reagents and Supplies

Reagent chemicals, purchased from reputable laboratory supply companies, are of ACS reagent grade or better. Calibration standards are either prepared in-house from high purity starting materials or purchased as standard concentrates. Commercial standards are certified by the supplier and are checked on receipt against the previous laboratory standard. Carrier gases, solvents, acids and deionized water are checked on a daily basis.

## SECTION V

### GENERAL QUALITY CONTROL PRACTICES

#### A. Blanks

##### 1. Method Blanks

Method blanks are introduced on a daily basis or at a frequency of one-in-twenty if more than that number of determinations are run in one day. The blanks consist of organic-free or deionized water which is then carried through the analytical scheme as for real samples. They serve to locate any contamination associated with laboratory storage, laboratory instrumentation, or equipment coming into contact with the sample during processing. They provide the fundamental baseline against which the sample signal is measured.

##### 2. Field and Travel Blanks

These closely related types of blanks find their most extensive application in analysis for volatile organic compounds. For each kind, the blank begins as organic-free reagent water in the laboratory. For a travel blank, a sample vial is filled with the reagent water at the laboratory and then carried with the sample containers out to the field and back to the laboratory. Field blanks are carried to the sample site in a separate container, with a vial being filled at the sampling location. In either case, the blank serves to identify and possibly correct for contamination associated with collecting or transporting the sample.

##### 3. Sample Blanks

These blanks come into play when native sample characteristics (most often color or turbidity) interfere with a determination. If the method in question relies on spectrophotometric detection, the sample's original absorbance at the wavelength of interest is measured and subtracted from the absorbance of the final developed color. The value is recorded in the bound bench book so both the apparent concentration and the corrected value may be calculated.

## B. Standard Curves

Spectrophotometers are standardized daily with at least three concentrations for every test. Absorbances are recorded in the bound notebook associated with each test; this step permits detection of any change in absolute performance. Indications of declining sensitivity or wavelength inaccuracy are corrected by having the unit professionally serviced.

Gas chromatograph and GC/MS determinations are standardized with curves containing at least three points as described in the October 26, 1984 Federal Register. The curves are verified on a daily basis with a quality control standard.

Standard solutions are prepared at intervals specified in the referenced methods. Where no limit is stated, they are replaced at intervals short enough to prevent detectable deterioration during the lifetime of the material. Even when no deterioration is detected, standards are replaced at least once every six months.

## C. Internal Standards and Surrogates

Internal standards are added to concentrated sample extracts of semivolatile priority pollutants and to the purging aliquots of volatile priority pollutant samples. They serve to detect losses during capillary column injection of semivolatiles or during the purging of volatile priority pollutants. They provide a means for applying corrections to individual sample results providing that such losses are not extraordinarily large.

Surrogates are typically spiked into the samples prior to extraction and thereby provide recovery data for sample workup. The recovery data can be used to identify systematic recovery problems or sample specific extraction problems.

#### D. Duplicates and Spikes

Every tenth sample is analyzed in duplicate. The duplicate aliquots are carried through the entire workup and analytical scheme. The automated log-in system on the computer assigns samples for duplication based on designated matrix type. This assures that samples such as soils and hazardous wastes are duplicated at least as frequently as waters and wastewaters. How closely the results compare with each other provides a measure of precision for the determination.

The same sample which is duplicated is also subjected to a spike analysis. In this technique, a third aliquot of the sample has added to it a known quantity of the analyte. The recovery on the resulting spiked sample relative to its theoretical value reflects the accuracy of the determination. Since percent recoveries are often strongly influenced by the sample matrix, spiking real samples provides information on interferences as well as method performance.

Duplicate results and spike recoveries are sorted by matrix type for statistical analysis and calculation of control charts. If a particular determination is not carried out frequently enough on a particular matrix type for successful statistical manipulation, the results are grouped with others of similar matrices.

#### E. Laboratory Control Standards

These standards are purchased or prepared independently of method calibration standards. For every ten samples logged in for a particular determination, the computer assigns a laboratory control standard (LCS). The true value and the recovered concentration are archived along with duplicate and spike results.

## SECTION VI

### SPECIALIZED QUALITY CONTROL PROCEDURES

#### A. Organics by Gas Chromatography

Specialized quality control procedures for these analyses are typified by those used for Pesticides and PCB by EPA Method 608. For every nine samples analyzed, another three QC samples--a method blank, a duplicate, and a spike--are run. One QC control sample is analyzed for every 10 client samples. The results are compared to the acceptance criteria listed by EPA in the Federal Register. If the result for a particular constituent does not meet the criteria, the sample is reanalyzed for that constituent.

#### B. Organics by GC/MS

##### 1. Volatile Organics; EPA Method 624

Prior to the analysis of samples, and after meeting the calibration criteria for 50 ng Bromofluorobenzene (BFB), the GC/MS system is initially calibrated at a minimum of five concentrations to determine the linearity of response. Linear calibration curves and response factors for each of the EPA Method 624 compounds are periodically established by analyzing five concentrations over a 50 to 200 ug/L range. These response factors are verified every twelve hours by analyzing a QC check sample. Daily operation also includes tuning the GC/MS system with BFB to meet instrument performance criteria established by the EPA. For every nine samples analyzed, a method blank, a duplicate, and a spike are also run. Three surrogates and three internal standards are added to each sample in order to monitor purging efficiency and instrument operation.

2. Base/Neutral and Acid Extractable Organics; EPA Method 625

Before any samples are analyzed, the GC/MS system must be tuned to meet the ion abundance criteria for a 50 ng Decafluorotriphenylphosphine (DFTPP) sample every twelve hours. Five-point calibration curves for each of the EPA Method 625 parameters are established periodically. The corresponding response factors are verified every twelve hours by analyzing a QC check sample. Daily ion abundance criteria are met by tuning the instrument against a DFTPP standard. Five surrogate standards are added prior to extraction of the sample in order to monitor the extraction efficiency of the method. A daily sensitivity check is done by adding six internal standards in each sample extract. In addition, three quality control samples, a method blank, a duplicate and a spike are analyzed every batch of nine samples.

C. Metals Analysis

Most metals analyses are done by one of three techniques: flame atomic absorption spectrophotometry (FAA), graphite furnace atomic absorption spectrophotometry (GFAA), or inductively coupled argon plasma emission spectrophotometry (ICP). Available supplementary techniques include cold vapor and hydride generation. All sample digestions follow EPA or Standard Methods prescribed procedures. A daily method blank is run for each element. A three-point calibration curve is determined daily for each element. Calibration standards for that curve are prepared by fresh dilution of 1000 mg/L certified standards obtained from commercial sources.

When the concentration of the metal being determined exceeds the highest standard, the sample is diluted so it falls within the range of calibration. A daily laboratory control standard is run for all metals. The accuracy of analysis of metals in soils is checked periodically by analyzing a National Bureau of Standards reference material such as SRM #1646, an estuarine sediment.

## D. Selected General Chemistry Procedures

### 1. Biochemical Oxygen Demand (BOD)

Samples for five-day BOD are stored at 4 degrees C and set within 48 hours of receipt. To test the quality of the dilution water, its dissolved oxygen is measured initially and at the end of the five-day incubation period. Three dilutions of each sample are set. The oxygen depletion of at least one of the dilutions must be at least 2 mg/L; the final dissolved oxygen content must be greater than 1 mg/L. One duplicate and one glucose-glutamic acid laboratory control standard are set with each batch of samples. Fresh BOD seed in the form of settled primary effluent from a municipal wastewater treatment facility is obtained weekly. Seed material for analysis of industrial samples is also available.

### 2. General Mineral Analysis

Several checks on the accuracy of the analysis of drinking water for general minerals are applied. The anion-cation balance must agree to within  $\pm 3$  percent. Also, the total dissolved solids content should fall within 65 to 75 percent of the specific conductance.

### 3. Colorimetric Analysis

Chemical oxygen demand, cyanide, phenol, nitrate, nitrite, and phosphate can all be done colorimetrically. Each analysis requires a three-point calibration. A linear regression is performed to ensure that operating conditions are in order, and that the analyst is working in the linear range. Typical values for the correlation coefficient exceed 0.995.

### 4. Titrimetric Analyses

Hardness, alkalinity, chloride, free CO<sub>2</sub>, and chemical oxygen demand can all be determined titrimetrically. Titrants are standardized using primary standards. Deterioration of the titrant is avoided through monthly checks of the standardization. Careful review of laboratory control standards will also assist in identifying gradual changes in normality.



## 5. Gravimetric Analyses

Oil and grease, total solids, dissolved solids, suspended solids and gravimetric sulfates all fall in this category. Each analysis depends heavily on the accuracy of the balance used. For this reason, balances are checked each week against Class "S" weights. Dessicants are monitored to ensure a "moisture free" environment during storage of samples. Oven temperatures are also monitored regularly to ensure compliance with those specified in the EPA Methods.

### E. Microbiology

Certification requirements of the California Department of Health Services control the principal features of microbiology QC. These features include daily recording of all incubator temperatures, recording and filing of the autoclave performance record, sterilization of sample containers, application of a dechlorinating agent to sample containers, and monthly performance of a completed coliform test to verify routine confirmed coliform results.

An annual water suitability test is run to make sure the purified water used in media preparation contains no growth promoting or inhibitory substances. An inhibitory residue test is carried out annually on glassware to verify that routine cleaning procedures will not adversely affect results.

### F. Fish Bioassay

Fish toxicity bioassays are carried out according to California Department of Fish and Game Guidelines, using specified test organisms, sample dilutions, and sample volumes. They are conducted in a constant temperature room in which fish are acclimated for seven days prior to use in tests. Checks performed before and during tests on the control tank and on all dilutions are pH, temperature, and dissolved oxygen level. Freshwater bioassays are checked for alkalinity and hardness while saline or brackish bioassays are checked for conductivity at the beginning of the test. A test is considered invalid if there is more than 10 percent mortality among control fish during the test. Temperatures of test solutions must be within the specified test range for the organism used. Confidence limits are established using procedures provided by California Department of Fish and Game.

SECTION VII  
PERFORMANCE AUDITS

A. Laboratory Control Standards and Check Samples

Certified reference materials are acquired from the National Bureau of Standards for metals in tissue and sediment-like matrices. EPA provides performance evaluation standards for both metals and organics. Additional certified reference materials are provided by the American Industrial Hygiene Association as part of their laboratory certification program. Using external sources for control standards ensures uniform and accurate results.

B. Certification Programs

The laboratories are subjected to performance audits initiated several times every year. Recent audits have included:

1. USEPA semiannual drinking water performance check samples (WS series).
2. USEPA semiannual wastewater performance check samples (WP series).
3. California Department of Health Services (DOHS) certification for the analysis of priority pollutants and agricultural pesticides in well water, 1984.
4. Arizona Department of Health Services certification for the analysis of Title 22 constituents and fumigants (EDB, DBCP) in drinking water.
5. Orange County Environmental Management Agency approval to analyze for metals, inorganics, halogenated pesticides and PCBs.
6. California DOHS certification for complete chemical analysis of hazardous waste.

C. Brown and Caldwell Interlaboratory Comparisons

The exchange of samples between laboratories is a widely recognized quality assurance measure which provides information about procedural errors, contamination unique to a particular laboratory, and interlaboratory precision and accuracy. Since Brown and Caldwell Laboratories consists of two independently operated but similarly equipped laboratories, interlaboratory studies may be carried out with particular convenience. In this way one laboratory can serve as the external quality assurance unit for the other. The Sample Control Section of each laboratory is responsible for the splitting of samples for such comparative purposes. The interlaboratory exchange program is usually reserved for major projects with special QA/QC requirements.

D. Round Robin Studies

Brown and Caldwell Laboratories frequently participates in studies of methods or performance among groups of well qualified environmental laboratories. Samples for such studies have been provided by Los Angeles County Sanitation Districts, Orange County Sanitation Districts, Electric Power Research Institute, and other public agencies and trade associations.

## SECTION VIII

### DATA HANDLING AND ASSESSMENT

#### A. Computer Management

The laboratory utilizes a computerized sample control and data management system for collecting and reporting analytical data. Upon receipt, a sample is logged into the computer to initiate the process. Associated with each sample is a unique log number, client sample description, sample matrix type, required analyses, and due date. Also included is supplementary information such as special handling requirements.

The next morning, each section supervisor receives a packet of computer generated work sheets listing work in house arranged according to analytical parameter and due date. The sheets listing groups of samples are then distributed to the appropriate analysts. The analyst performs the necessary analysis and records the raw data in a permanently bound notebook. Calculations are made and the final result is entered onto the appropriate computer work sheet.

That evening, the data input operator enters the day's results into the computer where they are transferred to "work awaiting approval" (WAA) status. The following morning the computer prints the previously entered results on work sheets which each analyst checks against the original data as recorded in the analytical notebook for that parameter. The analyst initials the entry to signify approval or can make corrections if necessary.

Later that day the initials are entered into the computer, which then transfers the data to the "report" file. When all the results for a particular sample have been entered and approved, a final report is printed. The printed report is reviewed by the section supervisor and signed by the Laboratory Director before being mailed to the client.

This computerized system maintains a complete audit trail for the work done on each sample. Information such as date sampled, date received, analyst's name, date completed, and the analytical method used in each determination are all retrievable from the data base.

## B. Archiving

Following analysis, all samples are kept for a minimum of 30 days. In this way, questions raised during the review of data can be addressed by inspection of the sample or by complete reanalysis using a different method. Analysis for components subsequently added to the list of constituents to be measured and incompatible with previous sample workup may be analyzed without resampling. The chief quality assurance feature provided by the archived samples is the ability to use the archived samples to resolve problems which may be noted only as the data are compared and interpreted.

Organic sample concentrates are sorted by project, clearly labeled and stored in a freezer for future reanalysis. Therefore, if methodological advances, changes in scope, or evidence of instrument malfunction are developed following analysis, complete reprocessing of the sample is not necessary.

Raw data for most procedures are kept in bound notebooks associated with the test or group of determinations. The completed notebooks are filed in the laboratory for reference should future comparisons be desirable. Raw chromatographic data, such as that collected in the quantitation of trace organics, are filed to permit critical reexamination at any time. Such information is particularly useful in cases where the inspection of old data may yield clues regarding the presence of newly identified species.

GC/MS chromatograms are similarly archived together with the spectra of chromatographically isolated but unidentified components. In addition, raw GC/MS data are transferred to tape in an EPA approved format in order to permit reprocessing to retroactively search for new classes of compounds or otherwise reexamine previously reported findings.

In addition to computer storage, a hard copy printout of every sample report is filed under the client's name and saved for five years.

VIII-2

### C. Detection Limits

The method detection limit is often defined as the minimum concentration of analyte which can be identified, measured, and reported with 99 percent confidence that the concentration is greater than zero. The method detection limit is mathematically defined as three times the standard deviation of seven replicate analyses of the analyte in question. The seven replicate determinations must be nonconsecutive and the analyte must be within five times the estimated detection limit.

When a sample is diluted, the reported detection limit is equivalent to the method detection limit multiplied by the dilution factor. This procedure for establishing the method detection limit is provided in Appendix B of 40 CFR, Part 136, as published in the Federal Register of October 26, 1984.

### D. Assessment

Before the significance of analytical data can be assessed, it is necessary to know how precise, how accurate, and how complete the data subsets are. Precision is amenable to strict definition by the analysis of replicate results according to schemes outlined in the USEPA Handbook for Analytical Quality Control in Water and Wastewater Laboratories, March, 1979, Chapter 6. Accuracy is somewhat more difficult to assess. Spike recovery determinations, regular analysis of laboratory control standards, and use of external check samples contribute to the general assurance that the accuracy of a determination is within acceptable limits. The ultimate accuracy of a determination also depends on factors external to the laboratory such as sampling and storage conditions. Completeness is the most difficult characteristic to assess, depending as it does upon such factors as representativeness of sampling and subsampling, selection of the appropriate analytical constituents, and scope of the sampling program relative to the size of the environmental question being addressed.

While ultimate assessment depends on the experienced judgment of knowledgeable individuals, statistical treatment of the data can provide some objective measure of their soundness. The Federal Register includes required calculations for accuracy on spiked sample for

several organics methods. The same calculation may be used for any test amenable to spiking:

$$P = 100(A - B)/T$$

Where:

- P = Percent spike recovery
- A = Concentration determined on spiked sample
- B = Concentration determined on original unspiked sample
- T = True value of spike added

Precision values may be calculated from analysis of duplicate pairs. In his manual Quality Assurance of Chemical Measurements, John K. Taylor of the National Bureau of Standards provides a formula for calculating the standard deviation based on a series of duplicates. When a sufficient number of spiked samples or duplicate pairs (at least 20) have been analyzed, control charts may be calculated. Because of the wide diversity of sample types and many different determinations, control charts have usually been calculated only in connection with special programs requiring them. However, the laboratory computer system is now being programmed to calculate and maintain charts for every test on every type of sample. For accuracy, the mean recovery and standard deviation (S) are calculated.

## SECTION IX

### CORRECTIVE ACTION

The control chart, predetermined acceptance limit, or EPA acceptance criteria serve as alert systems for unsatisfactory or unexpected results. The nature of corrective action may take several forms, but the first step is usually to repeat the analysis on the sample which failed. If the repeat does not replicate the failure, and prior and subsequent QC data do not indicate a systematic error, the value may be treated as a random error and disregarded.

More commonly, diagnosis and correction of an analytical problem will follow. If the repeat analysis continues to show difficulty, the analyst will bring it to the Section Supervisor's attention. If the required correction is not readily apparent, the supervisor will call in the laboratory Technical Director and the Quality Assurance Director. Together, they plan a series of steps to isolate and correct the problem. Based on frequency of occurrence and correctibility, the usual checking order includes:

1. Check the calculations.
2. Check laboratory control standard. This may reveal systematic errors.
3. Examine the sample for non-homogeneity or peculiar interferences.
4. Check instruments for proper performance.
5. Verify that standard solutions are fresh and properly prepared.
6. Assure the purity of reagent water or reagent gases.
7. Closely observe the analyst to be certain no procedural errors are occurring.

During the troubleshooting process, routine analysis for that determination is discontinued. Once the problem is found and corrected, the best estimate is made of when the problem first occurred. Data collected after this critical point is discarded. If possible, all analyses since the last valid control check will be repeated. Analyses performed after the resolution of the problem must be accompanied by more



duplicates and spikes than the regular ten percent level. The higher level of QC continues until the Section Supervisor and the Quality Assurance Director are satisfied that the problem has been completely solved.

To assist efficient resolution of problems, the Quality Assurance Director maintains a test-based file of previous corrective actions. The material is readily available to both laboratories. Since some determinations may be subject to common errors, this program helps reduce the time required to correct a problem if the other division laboratory has previously dealt with the same issue.

## SECTION X

### LABORATORY MAINTENANCE

Service contracts are maintained for the Finnigan 4000 mass spectrometers. A spare parts inventory is maintained which includes heaters, sources, oven assemblies, filaments, traps, electronics modules, RF generators, power controllers, vacuum controllers, columns and jet separators. A service contract is also maintained for the Varian 3400 with ECD detector. Spare septa, columns (DB608 and DB5) and column fittings are kept. Maintenance on each instrument is recorded in log-books accessible for review.



HARGIS + ASSOCIATES, INC.

**APPENDIX C-2**

**LABORATORY QUALITY ASSURANCE/QUALITY CONTROL PLAN  
ANALYTICAL TECHNOLOGIES, INC.**



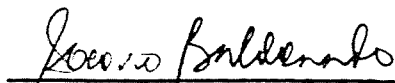
LABORATORY  
QUALITY ASSURANCE/QUALITY CONTROL PLAN

Approvals By:



Richard M. Amano  
ATI Project Manager

Date: 11/11/86



Spoorro R. Baldonado  
Laboratory Quality Assurance Officer

Date: 11/11/86

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## 1.0 INTRODUCTION

This project plan summarizes the Analytical Technologies, Inc. (ATI) quality assurance procedures in providing service as an analytical chemistry laboratory for government agencies such as EPA as well as commercial clients. The purpose of this plan is to assure that consistently accurate data are being reported in support of ATI's responsibilities as an environmental chemistry laboratory serving both government agencies and private sector concerns.

### 1.1 QA/QC Objectives

Overview: The major responsibility of ATI is to provide data which are timely, cost-effective, and of a quality required by the program. The accomplishment of these goals is the principal objective of ATI QA program. It recognizes that the data by our staff must be credible. This is ensured by the QA/QC program which is monitoring all phases of data generation, ranging from sample collection to sample handling, to the actual analysis and reporting of data.

### 1.2 QA/QC Scope and Approach relating to measurement of data in terms of precision, accuracy, completeness, representativeness, and comparability

1. ATI routinely checks the quality of analytical work through analysis of reference samples, duplicate samples, and spiked samples.

2. The accuracy of measurement data is evaluated by comparison of the % recovery of the reference material of known or established concentration, independent of routine calibration. It is used as prepared or diluted with inert matrix as a blind environmental sample. Statistically based control limits are established for each method of analysis and sample matrix.

3. Precision is routinely evaluated based upon the results of samples which are analyzed in duplicate. A duplicate sample is analyzed for each batch of ten samples (10%) for in-house QC. Small sample lots are routinely analyzed and data are reported on a frequent basis. The basic precision statistics from multiple small lots of a given sample matrix may be compared to develop a graphic assessment for the sample type.

4. The precision and accuracy is dependent on type of analysis, the sample matrix, and the concentration range of the particular analytes.

5. Spiked samples are run for each batch of 10 samples (10%) and/or each type of sample matrix. Recoveries are assessed to determine method success and interference effects.

6. All data are visually checked for consistency and reasonableness, any grossly high or grossly low results are checked. Data are calculated and reported in units consistent with the approved method for comparability of results. Unusually high or unexpectedly low results are verified using different methods where possible.

7. To evaluate completeness of extensive monitoring programs.

8. Upgrade the over-all quality of laboratory performance.

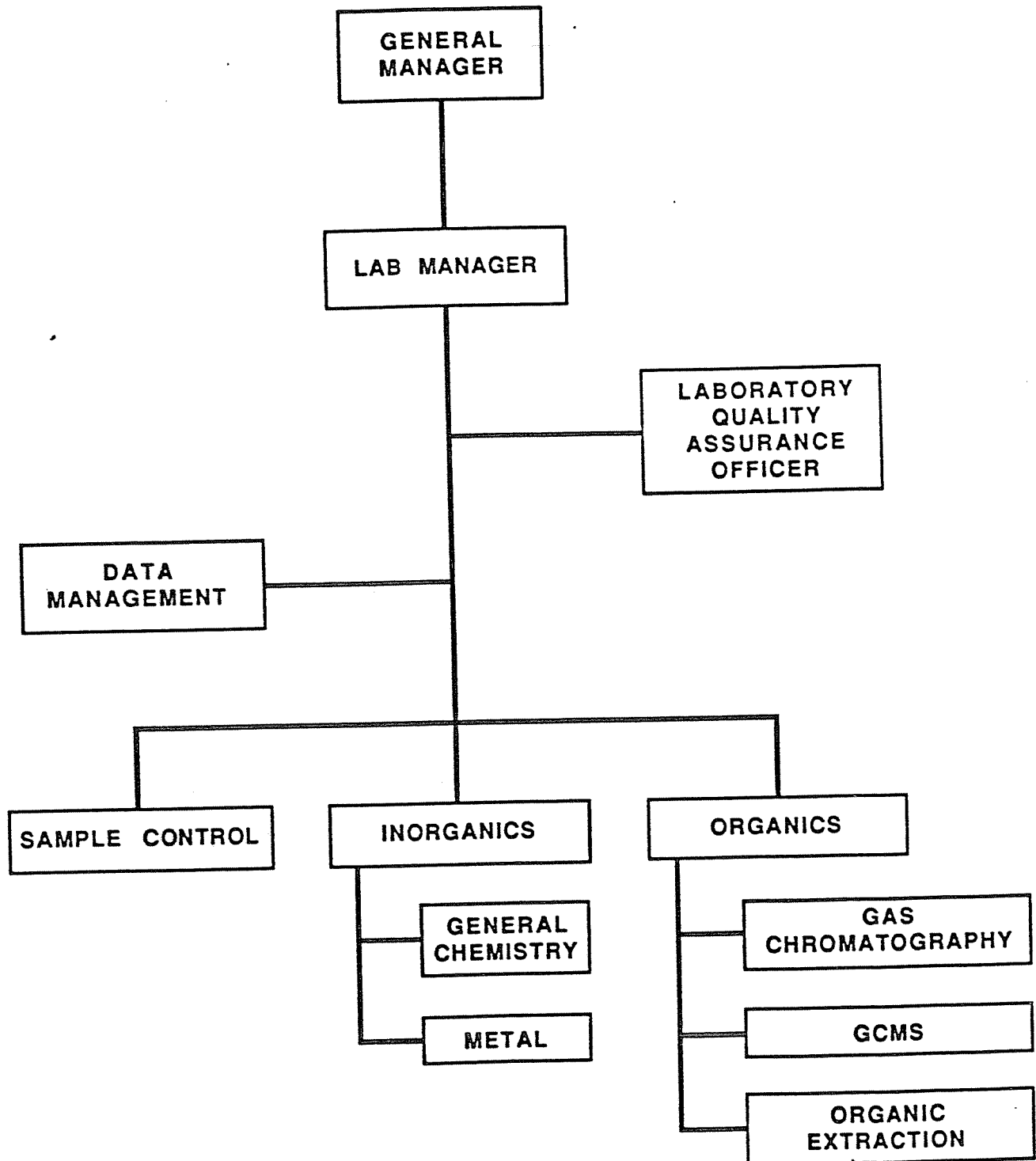


## 2.0 LABORATORY ORGANIZATION AND PERSONNEL

### 2.1 Overview

The following is a description of the operational structure of ATI's laboratory staff as it relates to responsibilities and functions in the QA/QC plan, with special focus on the Laboratory Quality Assurance Coordinator (LQAC). An organization/chain of command chart and a description of each key positions role is included.

# ATI, ORGANIZATION CHART



## 2.2 Roles and Responsibilities

### 2.2.1 The Laboratory Quality Assurance Coordinator

The Laboratory Quality Assurance Coordinator (LQAC) has responsibility for monitoring the quality of the laboratory's work and taking appropriate actions to ensure that quality standards are being met, including stopping the release of data which are of suspect quality. The LQAC has a high degree of independence and authority in the laboratory's organization. The LQAC reports directly to the laboratory manager to review the work of groups and individuals, and is generally independent of production pressures.

The LQAC carries out the following activities within the laboratory's operations:

- \* overseeing proficiency testing for laboratory approval, including handling of samples;
- \* coordinating any on-site QA/QC inspections;
- \* preparing or overseeing the preparation of the laboratory QA/QC plan;
- \* establishing QC procedures, providing control samples, and setting warning and action limits for every test;
- \* monitoring compliance with the laboratory's QA/QC plan:
  - reviewing QC related activities and documentation for completeness in accordance with the QA/QC plan;
  - identifying and referring any instances in which QC objectives are not being met to the section heads or laboratory manager for remedial action;
  - assuring that suspect data are not included in laboratory reports;
  - following up on the remedial actions undertaken in response to the above referrals to assure that QC objectives are once again being met.
- \* serving as laboratory point-of-contact for exchange of QA/QC information and approving, along with the Laboratory Manager release of QA/QC information;

- issuing the laboratory QA/QC plan;
- reporting changes in the QA/QC plan;
- issuing a corrective action plan in response to deficiencies identified during laboratory inspection;
- issuing corrective action reports for out-of-control events;
- responding to inquiries regarding laboratory QA/QC related activities;
- issuing the final QA/QC report.

#### 2.2.2 Other Laboratory Personnel - QA/QC Responsibilities

1. General Manager: The General Manager is responsible for the entire laboratory operation i.e., financial, technical and marketing, and accomplishes this function through delegation of tasks and supervision of subordinates. The General Manager has no direct QA/QC function, and the LQAC reports to this office through the Laboratory Manager.

2. Laboratory Manager: The person is in charge of all technical laboratory operations, which includes sample control, inorganic analysis, organic analysis, data management, and QA/QC. The Laboratory Manager directs the activity of the personnel in each operational section through the supervisors, to insure the QC procedures are being performed and any out of control situations or discrepancies are remedied properly and promptly.

3. Sample Custodian: The QA/QC responsibility here is very vital in that this person must maintain integrity and validity of the samples. The sample custodian insures that all samples are received in good condition in the proper containers/preservatives, are logged in correctly and accurately, chain of custody procedure is followed, and all samples are stored in secure areas under the correct temperature, light protection, and segregation (VOA samples) conditions. The sample custodian also supervises the cleaning of sample containers and laboratory glassware.

4/5. Inorganic and Organic Laboratory Supervisors: These individuals role in QA/QC is to insure that all analyses are conducted with the requisite QC parameters, instrument calibration and maintenance procedures are being followed and that all reagents, calibration materials, lab glassware, solvents, and other material used in analytical methods are of adequate quality and quantity.

6. Data Management Supervisor: The QA/QC function in this section is to maintain the day to day operations of data handling to ensure that clerical errors are kept to the very minimum and that all analytical data and QC data are properly collated into the reports. This individual is also responsible to make sure that all documentation is stored securely and is easily retrievable.

### 3.0 SAMPLE COLLECTION, PREPARATION, AND TRACKING

The objective of sampling is to collect a portion of material small enough in volume to be conveniently transported to and handled in laboratory while still accurately representing the materials being sampled. This implies, that the concentrations of all pertinent components must be the same in the sample as in the material being sampled, also that the sample must be handled in such a way that no significant changes in composition occur before the tests are performed.

#### 3.1 Sample Collection

Analytical Technologies, Inc. does not have the in-house capabilities of sample collection. Sampling is done by outside contractors and mostly by clients, i.e. EPA, and Environmental Engineering Consultants.

#### 3.2 Sample Preparation

For most commercial clients, ATI prepares all sample containers, including transport blanks, used in accordance with the requirements stated in the proposed rules 40 CFR, Part 136, Guidelines Establishing Test Procedures for the Analysis of Pollutants. Table II (see Appendix I for the ATI table representing test parameters, type of sample containers, preservation, and storage methods).

#### 3.3 Sample Tracking

Samples received at Analytical Technologies, Inc. are considered to be physical evidence and are handled according to procedural safeguards established by the EPA. The Sample Control Standard Operating Procedures (SOP) manual describes in detail how and where samples are received; the step-by-step sample log-in process; how samples are tracked - from receipt to completion; and the overall responsibilities of the sample custodian.

##### 3.3.1 Sample Verification and Log-In

After a sample shipment has arrived, the sample custodian will begin sample inspection and log-in, using ATI's Sample Condition and Verification Log form. This form serves as both a check-off list of procedures to follow and as documentation of the following:

1. Presence/absence of custody seals or tapes on the shipping containers and the condition of the seals (i.e. intact, broken);

2. Presence/absence of chain-of-custody;
3. Presence/absence of sample tags and if the tags are removable;
4. Agreement/non-agreement between the sample tags, chain-of-custody, and request forms; and
5. Condition of the samples when received e.g. cold or ambient, intact or broken/leaking, headspace in VOA vials, etc.

During sample receipt, the sample custodian will have checked and documented any problems or discrepancies with the sample shipment (see Sample Receipt). If the shipping containers were noted to be leaking, they should be moved to the hood and opened there.

Listed below is the step-by-step process for sample inspection:

1. Confirm that the airbill/bill of lading is present and record the airbill number.
2. Document the presence or absence of custody seals or tape on the shipping containers and the condition of the seals (i.e. intact, broken).
3. Open the shipping container and remove the enclosed samples and accompanying documents. If an odor is noticed after opening the shipping container, the VOA GC/MS operator should be notified.
4. Organize the sample bottles according to I.D. numbers. Document whether the samples were received:
  - a. cold or ambient
  - b. intact, broken/leaking
  - c. headspace in VOA vials
  - d. teflon septums inserted correctly
  - e. presence or absence of sample tags
5. Total the number of samples received and check agreement with the Sample Notification Form DC-1.
6. Confirm that the sample matrix agrees with the matrix cited on both Form DC-1 and the chain-of-custody.
7. Verify that the chain-of-custody and all forms have been received.
8. Document the agreement/non-agreement between the chain-of-custody, forms, and sample tags.

### 3.3.2. Sample Labelling System

Sample log-in begins by assigning an internal control number (ATI accession #). This number refers to a sample log-in book that is a permanent, sequentially numbered log of all batch samples received at Analytical Technologies, Inc. Recorded in the logbook is the ATI accession number, date of receipt, date report due, and the total number and type (matrix) of samples received. Each sample is assigned a unique number which includes its accession batch number. ATI accession numbering system is based on Julian calendar date system.

Example: 052 02

/ /

```

/===== refers to the Julian
/         calendar date.

```

```

/==== refers to the chrono-
logical sequence the
sample was log-in.

```

(see attached example)

### 3.3.3 Chain of Custody Procedures

### 3.3.3.1 Objectives:

Chain-of-custody procedures are utilized for a variety of samples received by the laboratory. The purpose of such procedures is to establish detailed legal documentation of all transactions in which the samples are transferred from the custody of one individual to another. These procedures are utilized from the point at which the samples are collected all the way to the opening of the sample in the laboratory and includes couriers that handle the samples. The importance of such evidentiary documentation cannot be strongly emphasized as it is possible that the document will be used in court cases or hearings.

### 3.3.3.2 Procedures:

1. Clients should utilize the chain-of-custody forms provided by the laboratory, or their own equivalent.
2. The client will retain the ORIGINAL and forward copies with the sample.
3. All information regarding the indentification of the client, the location of the



CLIENT: TempeACCESSION #: 05202  
DATE RECEIVED: 2-21-86

SAMPLE CONDITION UPON RECEIPT	YES	NO
1) Are EPA seals intact?	( ) <u>NA</u>	( )
2) Are EPA sample tags present?	( ) <u>↓</u>	( )
3) Is there a chain of custody?	(X)	( )
4) Are the sample tags, sample containers and chain of custody all in agreement? If no, explain _____	(X)	( )
5) Do the number of samples received agree with chain of custody? If no, explain _____	(X)	( )
6) Does the sample matrix agree with chain of custody? If no, explain <u>Not indicated</u>	( )	( )
7) Were the samples received cold?	(X)	( )
8) Are the sample containers intact? (i.e. not broken, leaking, etc.).	(X)	( )
9) Is there any headspace in the VOA vials? If so, which ones _____ <u>8 Water in 1 pt. Vocs per sample + 1 T.B.</u>	( )	(X)

## ATI SAMPLE I.D. (CROSS-REFERENCE WITH CLIENT I.D.)

1) <u>019401</u>	16) _____
2) <u>019403</u>	17) _____
3) <u>019404</u>	18) _____
4) <u>019405</u>	19) _____
5) <u>019406</u>	20) _____
6) <u>019407</u>	21) _____
7) <u>019408</u>	22) _____
8) <u>019409</u>	23) _____
9) <u>Trip Blank</u>	24) _____
10) _____	25) _____
11) _____	26) _____
12) _____	27) _____
13) _____	28) _____
14) _____	29) _____
15) _____	30) _____

sample, and the description of the sample, dates and times must be complete.

4. As the sample is transferred from the sampler to the courier(s) and then to the receiving department of the laboratory, signatures must be applied to the chain-of-custody forms both for the receiver and for the person releasing the specimen.

5. Each signature must be noted with the date and time of transfer.

6. DO NOT open the sample if any of the transfers have not been documented. Call the client immediately if exceptions are noted.

7. A copy of the completed form will be returned with the report.

3.3.3.3 Chain of Custody Record Example  
(see attached form)



Analytical Technologies, Inc.

San Diego, CA • Los Angeles, CA • Phoenix, AZ • Seattle, WA

### Chain of Custody Record

DATE \_\_\_\_\_ PAGE \_\_\_\_\_ OF \_\_\_\_\_

[illegible]

**DISTRIBUTION:** WHITE CANARY, ANALYTICAL TECHNOLOGIES INC., PRINC. ORIGINATOR

10 p

**BOE-C6-0183915**

#### 4.0 LIMITS OF DETECTION (LOD)

It is defined as the lowest concentration which can be detected with a specific level of confidence. It is the minimum concentration of a substance that can be measured and reported with 99% confidence that the true value, corresponding to a single measurement, is above zero.

ATI's methods for which the LOD are developed have been based on EPA methods for Organic Chemical Analysis of Municipal and Industrial Wastewater Appendix 4A - Definition and Procedure for the Determination of the Method Detection Limit (see Appendix IV).

As a participant of the EPA contract lab program, ATI follows the Contract Required Detection Limits (CRDL) established in the IFB manual (see Appendix III).

## 5.0 QUALITY CONTROL PROCEDURES AND DOCUMENTATION

### 5.1 Overview of Quality Control Program

#### 1. Quality Assurance\*

A quality assurance program is an essential part of a sound analytical protocol and should be used by individuals as well as by laboratory organizations to detect and correct problems in the measurement process or to demonstrate attainment of a state of statistical control. The objective of quality assurance programs for analytical measurements is to reduce measurement errors to agreed-upon limits and to assure that the results have a high probability of being acceptable quality. Two concepts are involved in quality assurance: (1) quality control, the mechanism established to control errors; and (2) quality assessment, the system used to verify that the analytical process is operating within acceptable limits.

#### 2. Quality Control\*

A quality control program includes the following: (1) development of and strict adherence to principles of good laboratory practice; (2) consistent use of standard operation procedures; (3) establishment of and adherence to carefully designed protocols for specific measurement programs; (4) the consistent use of qualified personnel; (5) reliable and well-maintained equipment; (6) appropriate calibrations and standards; and (7) the close supervision of all operations by management and senior personnel. When properly conceived and executed, a quality control program will result in a measurement system operating in a state of statistical control, which means errors have been reduced to acceptable levels and have been characterized statistically.

\* Principles of Environmental Analysis, Anal. Chem. 1983, 55, 2210-2213, L. Keith, et al.

### 3. Quality Assessment

This is the description of the techniques used to assess the quality of the measurement process and the results. The establishment of a system of "control charts" is a basic principle. Control charts are plots of multiple data points from the same or similar samples or processes versus time. They are used to determine if a system is in a state of statistical control. Control charts should be used to visualize or monitor the relative variability of repetitive reference materials, spiked samples, and the analysis of surrogates as a means of assessing the accuracy of measurements.

### 4. Completeness

It is a measure of the valid data obtained from a measurement system expressed as a percentage of the amount of data that should have been collected. Completeness is of particular importance to multi-year intensive monitoring programs.

At the end of a project or specified time period, calculate completeness as:

$$\text{Completeness, \%} = \frac{\text{Number of Valid Data Acquired}}{\text{Total Number of Values Planned}} \times 100$$

### 5. Blind Q.C. Samples (External/Q.C.)

These are samples submitted to our laboratory as ordinary soil, sediment, or water. Blind samples may include: (a) uncontaminated soil; (b) split samples; (c) unlabeled duplicates; and (d) performance evaluation samples.

### 6. QA Organization

Assembled data shall be reviewed by the appropriate section supervisor before technical compilation in deliverables package. Final review of the assembled deliverables package shall be undertaken by the project manager.

### 7. Standard Logs

"Standard" logs shall be maintained that shall indicate when prepared, concentrations, and by whom. They shall also indicate traceability back to the Primary Standard used. In addition, when a new lot of calibrators are prepared, a parallel testing chart shall record in current areas and response factors for the old lot side by side with a new lot. The intent is to provide maximum consistency of data.

## 8. Initial and Continuing Calibration Procedures

- a. Gas Chromatography Methods (GC), EPA Methods 601/8010, etc. Injection of secondary standards, validated by the use of EPA or NBS reference standards, are used to adjust the sensitivity and selectivity of the analytical system for each compound being analyzed. Calibration of the chromatographic system is accomplished by preparing standards at a minimum of three concentration levels for each analyte. The low level standard is at or near the established detection limit. The medium and high level standard are at concentrations that correspond to the expected range of concentrations found in real samples and will also define the working range of the GC detector. Standards are validated daily with the use of QC check solutions.

The results of standard calibrations (low, medium, and high ranges) for each analyte are tabulated with respect to response versus concentration. The ratio between response and concentration, known as response factor (RF), can be used to prepare a calibration curve for each compound. Alternately, if the RF is constant (less than 10% relative standard deviation) over the working range, linearity can be assumed and the average RF can be used in place of a calibration curve for each compound.

- b. Gas Chromatography/Mass Spectrometry (GC/MS), EPA Method 624/8240, 625/8270. Procedures for calibration and instrument tuning for sensitivity and selectivity are somewhat similar to those for gas chromatography methods. The primary difference between GC and GC/MS methods are concerned with the validation of the mass spectrometer as the detector. GC detectors generally operate by sensing a change in an electrical field, whereas, mass spectrometers sense a change in charge with reference to the mass of the compound. Further, the charged molecule ion will reproducibly fragment into an array of ions. The result is a characteristic mass spectrum of the compound. The first step in the calibration of the GC/MS system is to demonstrate the ionization and fragmentation of standard mass spectral tuning compounds. This is accomplished, as well as a sensitivity check, with the use of two EPA

specified (SW-846) compounds injected at concentration near the instrument detection limit, they are: 4 Bromofluorobenzene (BFB) for methods 624/8240 and Decafluorotriphenyl phosphine (DFTFP) for methods 625/8270 (see Ion Abundance Criteria - Tables 2 & 3). These standards are run daily to validate the GC/MS system tune.

Calibration of the GC/MS, like that of GC calibration, is established and validated by the injection of EPA traceable standards at a minimum of three concentration levels over the range of likely sample concentrations. An internal calibration procedure is used, that is, in addition to surrogate recovery compounds, sample extracts are spiked with internal calibration standards that span the retention time range of the analytes of interest. The concentration of the analytes are calculated with reference to the Response Factor (RF) of the internal standard for each sample.

Quality control is maintained by monitoring the responses of all of the parameters mentioned above on control charts. The procedures and control limits are specific by the method in SW-846. Further precision, accuracy and continuing calibration are demonstrated with the use of repeated analyses of spiked and unspiked samples and EPA check samples. Further, reagent blanks are analyzed in each batch of semi-volatile analyses (EPA 625/8270) and daily for volatile organic analyses (EPA 624/8240).

- c. Inductively Coupled Plasma (ICP) and Atomic Absorption Furnace (AAF), EPA methods 200.7/6010 and AA Furnace Series. Analytical Technologies, Inc. uses a combination of ICP, inductively coupled plasma, and AA, atomic absorption, for the greatest selectivity and sensitivity in the analysis of priority pollutant metals. These methods incorporate the differences in optical properties characteristic of the analytes in each of the methods.

Calibration standards, validate by reference standards, are prepared daily and monitored for stability periodically. As with most analytical techniques, the solutions are prepared at three different concentrations to demonstrate and verify the operating range of



the instrument. In addition to calibration standards, Quality Control is monitored by the use of analyses of Duplicate Samples, Spike Samples, QC-Check Samples, and Blank Samples every tenth sample. Calibration drift, within acceptable control limits, is verified and re-calibration is performed every 20 samples. Sample duplicates, spikes, and reagent blanks are carried through the whole analytical process.

Special attention is given to all samples with regard to interference, and appropriate action is taken to make corrections with the aid of ICAP interference check sample analysis. The ICAP interference check is analyzed at the beginning and end of each sample analysis run, or a minimum of twice per 8 hour working shift, whichever is most frequent. If an interference cannot be resolved successfully, standard addition technique will be used for both AA and ICAP. Standards of analytes to be determined will be added to the duplicate sample and concentration of the analyte(s) can be determined by difference.

## 9. Preventative Maintenance

A necessary adjunct to any quality control program is the development of a routine equipment maintenance program. Maintenance shall be required in response to failures encountered under the criteria section. It is required, however, to perform routine maintenance in an effort to reduce "down-time" on the equipment. Routine maintenance should adhere to a schedule, and any actions taken should be recorded. (see Appendix II for example).

### 5.2 Use of Control Samples and Reference Materials

#### 5.2.1 Type of Controls

1. Method Blanks (Reagent Blanks) - to check laboratory contamination.
2. Method Standards - to check the accuracy of the method under the optimum conditions excluding any chemical interference from sample matrix.
3. Duplicate Sample - to check laboratory precision.
4. Spiked Samples - to check recovery of

parameters of interest, including any chemical interference from sample matrix.

5. Reference Standards - used to check accuracy of both the digestion and the instrumental analysis. Standard reference materials are obtained from NBS and EPA.

6. EPA and/or commercial reference standards are analyzed weekly for the parameter of interest.

### 3.2.2 Preparation and Frequency of Use of Controls

#### 1. Duplicate Sample Analysis

A sample aliquot is taken through the whole sample preparation process. A duplicate sample is analyzed for each batch of ten samples (10%) for in-house QC.

Duplicate samples, like spike samples, are split at the very first stage of the analytical process. An equal aliquot of the sample is used. Like spike samples, water samples are mixed by shaking the sample container and measuring out equal aliquots. Soil samples are mixed with a stainless steel spatula in the sample jar or stainless steel mixing bowl and equal aliquots are measured out. Unlike field duplicates, precision is usually greater because the samples generally come from the same sample container.

#### 2. Method Spike Analysis

A duplicate sample aliquot is taken through the whole sample preparation process. Spike samples performed on 1 sample in each group of ten analyses, and/or each type of sample matrix. The only difference between the sample analysis process and the spike sample process is that after an aliquot of the sample is measured out, the substances that are to be analyzed are added in known amounts. The amount of spike added varies depending on the working range of the analytical instrument.

The value of the sample is determined, then the value of the sample spike is determined. Should the sample also have a value of the spiked analyte, the value of the sample is subtracted from the value of the spike and the percent (%) recovery of the spike is calculated using the following equation:

$$\% \text{ Recovery} = \frac{\text{Spike Sample Result} - \text{Sample Result}}{\text{Spike Added}} \times 100$$

At times the sample value is outside the operating range of the analytical instrument, in these cases it is impossible to know the magnitude of the analyte before the spike is added. Occasionally, the sample and the spike sample will require dilution to perform analysis of a particular sample within the dynamic range of the instrument. This dilution will serve to adjust the analyte in the sample to the proper concentration but will sometimes dilute the spike below the range of detection. The analyst in this case will not be able to report spike recovery for that particular analyte.

The lack of spike recovery data for an analyte that has been diluted to levels outside the working concentration of the instrument will be supplemented by the periodic analysis of spiked QC check sample and other additional sample data.

### 3. Method Blanks

Method blanks, also known as reagent blanks, are analyzed for each matrix and each batch of sample analyses. An aliquot, equal in volume or weight to the sample, of laboratory reagent grade deionized water is used for method blank analyses. The method blank, like that of duplicate and spike samples, is taken through the whole analytical process. Needless to say, the method blank should be blank of any substances being analyzed, or interferences.

### 4. Transport Blanks

Transport blanks, also known as trip blanks, are routinely analyzed while determining volatile organic compounds (VOA) in water. Whenever VOA containers are sent into the field for water sampling, they are always accompanied by a sample of "purgeable organics free water" which is returned to the laboratory and analyzed with the samples. This gives an insight into the possibility of contamination of VOA samples from outside sources. Likewise, transport blanks can be determined for any other testing parameters although outside sources of contamination are less likely.

### 5. Surrogate Spike Analyses

Where applicable, an analytical process includes the addition, subsequent detection, and recovery calculations of surrogate spiking compounds. Surrogate compounds are analyte compound substitutes, that is, they are compounds not specifically requested to be determined as analytes in a particular scope of work, and most often not naturally occurring. Surrogate compounds are added to samples for analysis after sample aliquots have been measured out and are taken through the whole sample preparation process. Surrogate compounds, to be useful in QC analysis, must not interfere with the determination of the analytes of interest. Surrogates must also be chemically similar to the analytes that are to be determined so as to

emulate the response of those analytes.

- a. Metal analysis does not have the luxury of additional QC via the use of surrogate spikes. There are no metals that can meet the criteria for surrogates as part of the QC regime.
- b. Pesticides/PCB analysis, EPA Method 608/8080, does not specifically identify the use of a pesticide/PCB surrogate spiking compound. However, ATI has adopted the EPA Contract Lab Program (CLP) guidelines for use of Dibutyl-chlorendate as a surrogate spike. The CLP has issued advisory limits for recovery of 24-154% for water analyses, and 20-150% for soil analyses.
- c. Volatile Organic Analysis (VOA), EPA Method 624/8240, is a GC/MS method. One of the advantages of using GC/MS is the reliability of the surrogate QC data. Using a mass spectrometer as the detector enables the selection of surrogates that are very similar chemically to the analytes of interest because the surrogates can be the same compound as the analyte varying only in molecular weight as a heavy isotope, e.g. Carbon-14 (C14) or Deuterium (D). For example, Toluene's (C7H8, MW=92) surrogate becomes Toluene D-8 (C7D8, MW=100). Only GC/MS methods have the ability to separate compounds by molecular weight, GC alone would not be able to distinguish between Toluene and Toluene-D8. For the complete list of surrogate spiking compounds, see Appendix 5.
- d. Semi-Volatile Organic Analysis (a.k.a Base/Neutral and Acid Compounds, BNA), EPA method 8270 is also a GC/MS method which incorporates the advantages of using a mass spectrometer and allows for use of heavy isotope labeled surrogates. Since this method is used to analyze compounds of varying acidity, the surrogates also vary across this spectrum. For the complete list of surrogate spiking compounds, see Appendix 5.

### 5.3 Establishment of Warning and Action Limits

Warning and action limits for control parameter have been established by a few different methods.

a) For organic parameters monitored in the EPA contract lab program, the limits have been set according to those established by the EPA.

b) For organic parameters not monitored in the EPA program, the limits have been set according to the Federal Register 40 CFR Part 136, October 1984.

c) For inorganic parameters, EPA guidelines have been used as criteria for the limits. The guidelines are for 95 percent confidence on known concentrations given to the laboratory.

d) Lastly, as the laboratory gathers a large enough data base, it will be able to establish its own criteria at a 95 percent confidence level.

#### 5.3.1 Approach to Setting Limits

1. The lab performs analytical instrument calibration check that covers the working range of methods being used.

2. A performance check mixture (solution) is used to demonstrate continued acceptable performance of the GC/MS/DS.

3. Concentration calibration solutions containing known amount of the analyte, the surrogate compound and the internal standard are used to determine instrument response of the analyte and the surrogate compound relative to the internal standard.

4. Analytical reference standards are analyzed as qualitative and quantitative checks on instrument performance during the course of analyzing samples.

5. The laboratory performs routine preventive maintenance to the instrument on a regularly scheduled basis.

6. Routine procedures are used to assess data precision, accuracy, and completeness.

#### 5.3.2 Documentation of Limits

All raw data and tabulated data used to verify or generate method detection limits (MDL) are held in files in the custody of the LQAC.

#### 5.4 Use of Control Charts

The performance of a measurement system can be demonstrated by the measurement of homogenous and stable control samples in a planned repetitive process. The data generated is plotted as a control chart to indicate whether the measurement system is in state of statistical control. It signifies the degree of replication of measurements of the control sample to provide confidence in the measurement process. It warns the laboratory of possible deviation from 95% confidence level by identifying systematic errors, drifts, or other types of problems.

#### 5.4.1 Types of Control Charts Used

Three charts will be used to monitor the data generated in the laboratory. The charts will be:

- a) Laboratory Control Standard (LCS) Concentration vs. Date Analyzed.
- b) Percent QA/QC Spike Recovery vs. Date Analyzed.
- c) Relative Percent Difference of Duplicates vs. Date Analyzed.

#### 5.4.2 Control Chart Preparation

The preparation of control chart is based on Shewhart's theory of control charts which are discussed in details in EPA Handbook for Analytical Quality Control Water and Wastewater Laboratories, Chapter 6.

#### 5.4.3 Approach to Control Chart Interpretation

1. For each parameter and method, a data base of %R for QC reference samples/or spiked samples is collected. The arithmetic mean and standard deviation of this set is calculated. From this information, warning and control limits of a run are determined.

Warning Limits are defined as  $\bar{x} \pm 2s$ , where  $s$  is standard deviation.

Control Limits are defined as  $\bar{x} \pm 3s$ , where  $s$  is standard deviation. The %R of each QC sample/or spiked sample is plotted on a control chart and compared with the statistically based control limits.

2. Data precision is evaluated based on the results of the samples analyzed in duplicate. The range is calculated and then divided by the average of the 2 analyses. When multiplied by 100, this values equals % difference. The % difference of duplicates in each data set is compared with the values previously found in the lab. Calculations for warning and control limits would be the same as the above.

3. Interpretation of control charts for out-of-control:

- one or more points outside the control limit (3s);
- a run of two or more points outside warning limits (2s);

- a run of seven or more points above  $\bar{x}$  or below  $\bar{x}$ , indicating trends or shifts; and
- cycles or non-random patterns in the data.



## 6.0 PROCEDURES FOR DETERMINING AND REPORTING OUT-OF-CONTROL EVENTS

### 6.1 Defining an Out-of-Control Event

An out-of-control event is defined as any occurrence failing to meet the QA/QC plan in relation to control chart interpretation. Control charts can reveal shifts, trends, biases, and conditions where parts of the analytical system are out-of-control. An out-of-control condition is related to the different zones on a control chart (e.g., data beyond the rejection limits, data in the zone(s) between the rejection and warning limits, and data inside the warning limits) and different patterns within these zones (e.g., number of consecutive data points on one side of the mean, number of consecutive data points in the middle zone, number of monotonically changing data points, obviously repetitive patterns; Garfield, 1984).

### 6.2 Responding to an Out-of-Control Event

#### 6.2.1 Roles and Responsibilities

When an out-of-control event is recognized, each individual involved with the analysis in question has an interactive role and responsibility, these are as follows:

- \* the analyst: He must be able to recognize QA failure and immediately notify the supervisor and work with the supervisor and LQAC to solve the problem; also maintains QC charts plotting.

- \* the supervisor: He must review all analytical and QC data for reasonableness, accuracy, and clerical errors; also responsible to monitor QC charts. In an out-of-control event, the supervisor works with the analyst and LQAC to solve the problem and prevents the reporting of suspect data by stopping work on the analysis in question and insuring that all results that are suspect are repeated, if possible, after the source of the error is determined and remedied.

- \* LQAC: In an event that an out-of-control situation occurs that is unnoticed at the bench or supervisory level, i.e. performance failure on a blind QC sample, the LQAC will notify the supervisor, help identify and solve the problem where applicable, insure the work is stopped on the analysis and no suspect data is reported. The LQAC must review and approve all corrective action reports, then submit them to the laboratory manager for review.

Lab ID: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

### OUT-OF-CONTROL EVENT REPORT

#### Description of Event

date recognized:  
date occurred:  
date corrected:

by:

by:

Analytical Method:  
Analyst:  
Supervisor:

Section:

Type of Event:

Why did event occur?

I.D. of Affected/Suspect Sample(s):

Corrective Action:

Signatures:

Supervisor:

Date:

LQAC:

Date:

Lab Manager:

Date:

### 6.2.2 Corrective Actions

This depends entirely on the type of analysis, the extent of the error, and whether the error is determinant or not. The corrective action to be taken is determined by either the supervisor, the analyst, the LQAC or by all three in conference, if necessary, but final approval is the responsibility of the LQAC and/or the laboratory manager.

A corrective action can be as extensive as replacing a complete lot of contaminated extraction solvent, re-extracting and analyzing a complete batch of samples, due to reagent blank contamination, or as simple as recalculating a series of results because a wrong dilution factor was applied. Again, the right corrective action must be determined on a case by case basis.

### 6.3 Documenting an Out-of-Control Event

This is accomplished by filling out an out-of-control event report form. This form is initiated either by section supervisor or the LQAC depending on what level the problem is recognized. The report will describe the analysis involved, the date, analyst, the identification of all affected or suspect samples, probable cause, the corrective action measure(s) taken and the final disposition/resolution of the problem. The report is to be signed by the section supervisor, the LQAC, and finally, the laboratory manager (see attached example form).

## 7.0 REFERENCES

1. Handbook for Analytical Quality Control in Water and Wastewater Laboratories, U.S. EPA 600/4-79-019, March 1979.
2. Federal Register, p. 43260, et seq., October 26, 1984
3. Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, U.S. EPA 600/4-82-057, July 1982.
4. Test Methods for Evaluating Solid Waste, U.S. EPA July 1982, 2nd ed., SW-846.
5. USEPA Contract Laboratory Program - Statement of Work for Organic Analysis, July 1985 revision (IFB WA85-J178).

# APPENDIX I

## REQUIREMENTS FOR SAMPLE CONTAINERS, PRESERVATION PROCEDURES AND MAXIMUM HOLDING TIMES OF SAMPLES

Test	Container <sup>(a)</sup>	Preservation <sup>(b)</sup>	Maximum Holding Time <sup>(c)</sup>
Acidity	P,G	Cool, 4°C	14
Alkalinity	P,G	Cool, 4°C	14 days
Ammonia	P,G	Cool, 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	14 days
<u>Biological</u>			
Coliform, fecal and total	P,G	Cool, 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> *	6 hours
Fecal streptococci	P,G	Cool, 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> *	6 hours
Biochemical oxygen demand	P,G	Cool, 4°C	48 hours
Bromide	P,G	None required	28 days
Chemical oxygen demand	P,G	Cool, 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
Chloride	P,G	None required	28 days
Chlorine, total residual	P,G	Determine on site	Immediately
Color	P,G	Cool, 4°C	48 hours
Cyanide, total and amenable	P,G	Cool, 4°C NaOH to pH>12 Ascorbic Acid*	14 days
Dissolved oxygen			
Proble	G bottle & top	Determine on site	1 hour
Winkler	G bottle & top	Fix on site	8 hours
Fluoride	P	None required	28 days
Hardness	P,G	HNO <sub>3</sub> to pH<2	6 months
Hydrogen ion (pH)	P,G	Determine on site	Immediately
Kjeldahl and organic nitrogen	P,G	Cool, 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days

\*required if residual chlorine present

# APPENDIX I

## REQUIREMENTS FOR SAMPLE CONTAINERS, PRESERVATION PROCEDURES AND MAXIMUM HOLDING TIMES OF SAMPLES (Continued)

Test	Container (a)	Preservation (b)	Maximum Holding Time (c)
Chromium VI	P,G	Cool, 4°C	24 hours
Mercury	P,G	HNO <sub>3</sub> to pH<2	28 days
Metals except above	P,G	HNO <sub>3</sub> to pH<2	6 months
Nitrate	P,G	Cool, 4°C	48 hours
Nitrate-nitrite	P,G	Cool, 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
Nitrite	P,G	Cool, 4°C	48 hours
Oil and grease	G	Cool, 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
Extractables (including phthalates, nitrosamines organochlorine pesticides, PCBs, nitroaromatics, isophorone, polynuclear aromatic hydrocarbons, haloethers, chlorinated hydrocarbons and TCDD)	G, teflon-lined cap	Cool, 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> *	7 days (until extraction) 30 days (after extraction)
Extractables (phenols)	G, teflon-lined cap	Cool, 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> *	7 days (until extraction) 30 days (after extraction)
Purgeables (Halocarbons and Aromatics)	G, teflon-lined septum	Cool, 4°C Add HCl to pH<2**	14 days
Purgeables (Acrolein and Acrylonitrile)	G, teflon-lined septum	Cool, 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> *	14 days
Organic Carbon	P,G	Cool, 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
Orthophosphate	P,G	Filter on site Cool, 4°C	48 hours

\*required if residual chlorine present

\*\*required for aromatics only

# APPENDIX I

## REQUIREMENTS FOR SAMPLE CONTAINERS, PRESERVATION PROCEDURES AND MAXIMUM HOLDING TIMES OF SAMPLES (Continued)

Test	Container <sup>(a)</sup>	Preservation <sup>(b)</sup>	Maximum Holding Time <sup>(c)</sup>
Phenols	G	Cool, 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
Phosphorus (elemental)	G	Cool, 4°C	48 hours
Phosphorus, total	P,G	Cool, 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
<u>Radiological</u>			
Alpha, Beta and radium	P,G	HNO <sub>3</sub> to pH<2	6 months
Residue, total	P,G	Cool, 4°C	7 days
Residue, filterable	P,G	Cool, 4°C	7 days
Residue, nonfilterable	P,G	Cool, 4°C	7 days
Residue, settleable	P,G	Cool, 4°C	48 hours
Residue, volatile	P,G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific conductance	P,G	Cool, 4°C	28 days
Sulfate	P,G	Cool, 4°C	28 days
Sulfide	P,G	Add NaOH to pH>9	7 days
Sulfite	P,G		Analyze Immediately
Surfactants	P,G	Cool, 4°C	48 hours
Temperature	P,G	Determine on site	Immediately
Turbidity	P,G	Cool, 4°C	48 hours

## APPENDIX I

### REQUIREMENTS FOR SAMPLE CONTAINERS, PRESERVATION PROCEDURES AND MAXIMUM HOLDING TIMES OF SAMPLES (Continued)

- (a) Polyethylene (P) or Glass (G).
- (b) Sample preservation should be performed immediately upon sample collection. For composite samples each aliquot should be preserved at the time of collection, if possible. Aliquots of the composite, which would require multiple preservatives, should be preserved only by maintaining at 4°C until compositing and sample splitting is completed.
- (c) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time.

Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample stability.
- (d) Samples should be filtered immediately on-site before adding preservation for dissolved metals.
- (e) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific organic compounds.



## APPENDIX II

### ANALYTICAL TECHNOLOGIES, INC. EQUIPMENT MAINTENANCE

INSTRUMENT MODEL Atomic Absorption 3030  
 MANUFACTURER Perkin Elmer SERIAL # 123655  
 DATE OF INSTALLATION Aug. 25, 1982 SERVICE PHONE # 714-544-8383  
 MFG. CONTACT PERSON Tony Rhoden

#### MAINTENANCE RECORD

DATE	TYPE	COMMENTS
10-1-85	A thru F	DG - several graphite tubes used daily
10-2-85	A thru F	DG
10-3-85	A thru F	DG
10-4-85	A thru F	DG
10-7-85	A thru F	DG
10-8-85	A thru F	DG
10-9-85	A thru F	DG
10-10-85	A thru F	DG
10-11-85	A thru F	DG
10-14-85	A thru F	DG
10-15-85	A thru F	DG
10-16-85	A thru F	DG had to repeat wheel because poor tube, adjusted auto sampler
10-17-85	A thru F	DG

#### MAINTENANCE TYPES

##### WEEKLY

- A. Inspect flush water supply and cooling water supply.
- B. Check furnace alignment and sensor windows.
- C. Replace graphite tube.
- D. Inspect lamps.

##### Daily

##### MONTHLY

- E. Clean contact cylinders.
- F. Test program.

##### QUARTERLY

- G. Lubricate auto sampler.
- H. Check power line conditions.
- I. Miscellaneous (insert comments)

# APPENDIX III

## Exhibit A (Sheet 1 of 6)

### Hazardous Substance List (HSL)\* and Contract Required Detection Limits (CRDL)\*\*

Volatiles	CAS Number	Detection Limits	
		Low Water <sup>a</sup> ug/L	Low Soil/Sediment <sup>b</sup> ug/Kg
1. Chloromethane	74-87-3	10	10
2. Bromomethane	74-83-9	10	10
3. Vinyl Chloride	75-01-4	10	10
4. Chloroethane	75-00-3	10	10
5. Methylene Chloride	75-09-2	5	5
6. Acetone	67-64-1	10	10
7. Carbon Disulfide	75-15-0	5	5
8. 1,1-Dichloroethene	75-35-4	5	5
9. 1,1-Dichloroethane	75-35-3	5	5
10. trans-1,2-Dichloroethene	156-60-5	5	5
11. Chloroform	67-66-3	5	5
12. 1,2-Dichloroethane	107-06-2	5	5
13. 2-Butanone	78-93-3	10	10
14. 1,1,1-Trichloroethane	71-55-6	5	5
15. Carbon Tetrachloride	56-23-5	5	5
16. Vinyl Acetate	108-05-4	10	10
17. Bromodichloromethane	75-27-4	5	5
18. 1,1,2,2-Tetrachloroethane	79-34-5	5	5
19. 1,2-Dichloropropane	78-87-5	5	5
20. trans-1,3-Dichloropropene	10061-02-6	5	5
21. Trichloroethene	79-01-6	5	5
22. Dibromochloromethane	124-48-1	5	5
23. 1,1,2-Trichloroethane	79-00-5	5	5
24. Benzene	71-43-2	5	5
25. cis-1,3-Dichloropropene	10061-01-5	5	5

# APPENDIX III

## Exhibit A (Sheet 2 of 6)

Volatiles	CAS Number	Detection Limits	
		Low Water <sup>a</sup> ug/L	Low Soil/Sediment <sup>b</sup> ug/Kg
26. 2-Chloroethyl Vinyl Ether	110-75-8	10	10
27. Bromoform	75-25-2	5	5
28. 2-Hexanone	591-78-6	10	10
29. 4-Methyl-2-pentanone	108-10-1	10	10
30. Tetrachloroethene	127-18-4	5	5
31. Toluene	108-88-3	5	5
32. Chlorobenzene	108-90-7	5	5
33. Ethyl Benzene	100-41-4	5	5
34. Styrene	100-42-5	5	5
35. Total Xylenes		5	5

<sup>a</sup>Medium Water Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 times the individual Low Water CRDL.

<sup>b</sup>Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 times the individual Low Soil/Sediment CRDL.

# APPENDIX III

## Exhibit A (Sheet 3 of 6)

Semi-Volatiles	CAS Number	Detection Limits	
		Low Water <sup>c</sup> ug/L	Low Soil/Sediment <sup>d</sup> ug/Kg
36. N-Nitrosodimethylamine	62-75-9	10	330
37. Phenol	108-95-2	10	330
38. Aniline	62-53-3	10	330
39. bis(2-Chloroethyl) ether	111-44-4	10	330
40. 2-Chlorophenol	95-57-8	10	330
41. 1,3-Dichlorobenzene	541-73-1	10	330
42. 1,4-Dichlorobenzene	106-46-7	10	330
43. Benzyl Alcohol	100-51-6	10	330
44. 1,2-Dichlorobenzene	95-50-1	10	330
45. 2-Methylphenol	95-48-7	10	330
46. bis(2-Chloroisopropyl) ether	39638-32-9	10	330
47. 4-Methylphenol	106-44-5	10	330
48. N-Nitroso-Dipropylamine	621-64-7	10	330
49. Hexachloroethane	67-72-1	10	330
50. Nitrobenzene	98-95-3	10	330
51. Isophorone	78-59-1	10	330
52. 2-Nitrophenol	88-75-5	10	330
53. 2,4-Dimethylphenol	105-67-9	10	330
54. Benzoic Acid	65-85-0	50	1600
55. bis(2-Chloroethoxy) methane	111-91-1	10	330
56. 2,4-Dichlorophenol	120-83-2	10	330
57. 1,2,4-Trichlorobenzene	120-82-1	10	330
58. Naphthalene	91-20-3	10	330
59. 4-Chloroaniline	106-47-8	10	330
60. Hexachlorobutadiene	87-68-3	10	330
61. 4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	10	330
62. 2-Methylnaphthalene	91-57-6	10	330
63. Hexachlorocyclopentadiene	77-47-4	10	330
64. 2,4,6-Trichlorophenol	88-06-2	10	330
65. 2,4,5-Trichlorophenol	95-95-4	50	1600

# APPENDIX III

## Exhibit A (Sheet 4 of 6)

Semi-Volatiles	CAS Number	Detection Limits	
		Low Water <sup>c</sup> ug/L	Low Soil/Sediment <sup>d</sup> ug/Kg
66. 2-Chloronaphthalene	91-58-7	10	330
67. 2-Nitroaniline	88-74-4	50	1600
68. Dimethyl Phthalate	131-11-3	10	330
69. Acenaphthylene	208-96-8	10	330
70. 3-Nitroaniline	99-09-2	50	1600
71. Acenaphthene	83-32-9	10	330
72. 2,4-Dinitrophenol	51-28-5	50	1600
73. 4-Nitrophenol	100-02-7	50	1600
74. Dibenzofuran	132-64-9	10	330
75. 2,4-Dinitrotoluene	121-14-2	10	330
76. 2,6-Dinitrotoluene	606-20-2	10	330
77. Diethylphthalate	84-66-2	10	330
78. 4-Chlorophenyl Phenyl ether	7005-72-3	10	330
79. Fluorene	86-73-7	10	330
80. 4-Nitroaniline	100-01-6	50	1600
81. 4,6-Dinitro-2-methylphenol	534-52-1	50	1600
82. N-nitrosodiphenylamine	86-30-6	10	330
83. 4-Bromophenyl Phenyl ether	101-55-3	10	330
84. Hexachlorobenzene	118-74-1	10	330
85. Pentachlorophenol	87-86-5	50	1600
86. Phenanthrene	85-01-8	10	330
87. Anthracene	120-12-7	10	330
88. Di-n-butylphthalate	84-74-2	10	330
89. Fluoranthene	206-44-0	10	330
90. Benzidine	92-87-5	50	1600
91. Pyrene	129-00-0	10	330
92. Butyl Benzyl Phthalate	85-68-7	10	330
93. 3,3'-Dichlorobenzidine	91-94-1	20	660
94. Benzo(a)anthracene	56-55-3	10	330
95. bis(2-ethylhexyl)phthalate	117-81-7	10	330
96. Chrysene	218-01-9	10	330
97. Di-n-octyl Phthalate	117-84-0	10	330
98. Benzo(b)fluoranthene	205-99-2	10	330
99. Benzo(k)fluoranthene	207-08-9	10	330
100. Benzo(a)pyrene	50-32-8	10	330

# APPENDIX III

## Exhibit A (Sheet 5 of 6)

Semi-Volatiles	CAS Number	Detection Limits	
		Low Water <sup>c</sup> ug/L	Low Soil/Sediment <sup>d</sup> . ug/Kg
101. Indeno(1,2,3-cd)pyrene	193-39-5	10	330
102. Dibenz(a,h)anthracene	53-70-3	10	330
103. Benzo(g,h,i)perylene	191-24-2	10	330

<sup>c</sup>Medium Water Contract Required Detection Limits (CRDL) for Semi-Volatile HSL Compounds are 100 times the individual Low Water CRDL.

<sup>d</sup>Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Semi-Volatile HSL Compounds are 60 times the individual Low Soil/Sediment CRDL.

# APPENDIX III

## Exhibit A (Sheet 6 of 6)

Pesticides	CAS Number	Detection Limits	
		Low Water <sup>e</sup> ug/L	Low Soil/Sediment <sup>f</sup> ug/Kg <sup>m</sup>
104. alpha-BHC	319-84-6	0.05	2.0
105. beta-BHC	319-85-7	0.05	2.0
106. delta-BHC	319-86-8	0.05	2.0
107. gamma-BHC (Lindane)	58-89-9	0.05	2.0
108. Heptachlor	76-44-8	0.05	2.0
109. Aldrin	309-00-2	0.05	2.0
110. Heptachlor Epoxide	1024-57-3	0.05	2.0
111. Endosulfan I	959-98-8	0.05	2.0
112. Dieldrin	60-57-1	0.10	4.0
113. 4,4'-DDE	72-55-9	0.10	4.0
114. Endrin	72-20-8	0.10	4.0
115. Endosulfan II	33213-65-9	0.10	4.0
116. 4,4'-DDD	72-54-8	0.10	4.0
117. Endrin Aldehyde	7421-93-4	0.10	4.0
118. Endosulfan Sulfate	1031-07-8	0.10	4.0
119. 4,4'-DDT	50-29-3	0.10	4.0
120. Endrin Ketone	53494-70-5	0.10	4.0
121. Methoxychlor	72-43-5	0.5	20.0
122. Chlordane	57-74-9	0.5	20.0
123. Toxaphene	8001-35-2	1.0	40.0
124. AROCLOR-1016	12674-11-2	0.5	20.0
125. AROCLOR-1221	11104-28-2	0.5	20.0
126. AROCLOR-1232	11141-16-5	0.5	20.0
127. AROCLOR-1242	53469-21-9	0.5	20.0
128. AROCLOR-1248	12672-29-6	0.5	20.0
129. AROCLOR-1254	11097-69-1	1.0	40.0
130. AROCLOR-1260	11096-82-5	1.0	40.0

<sup>e</sup>Medium Water Contract Required Detection Limits (CRDL) for Pesticide HSL Compounds are 100 times the individual Low Water CRDL.

<sup>f</sup>Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Pesticide HSL compounds are 60 times the individual Low Soil/Sediment CRDL.

## APPENDIX III

## INSTRUMENT DETECTION LIMITS

LAB NAME ANALYTICAL TECHNOLOGIES, INC.

<u>Compound</u>	<u>Required Detection Limits (CRDL)-ug/l</u>
Metals:	
1. <u>Aluminum</u>	200
2. <u>Antimony</u>	60
3. <u>Arsenic</u>	10
4. <u>Barium</u>	200
5. <u>Beryllium</u>	5
6. <u>Cadmium</u>	5
7. <u>Calcium</u>	5000
8. <u>Chromium</u>	10
9. <u>Cobalt</u>	50
10. <u>Copper</u>	25
11. <u>Iron</u>	100
12. <u>Lead</u>	5
13. <u>Magnesium</u>	5000
14. <u>Manganese</u>	15
15. <u>Mercury</u>	0.2
16. <u>Nickel</u>	40
17. <u>Potassium</u>	5000
18. <u>Selenium</u>	5
19. <u>Silver</u>	10
20. <u>Sodium</u>	5000
21. <u>Thallium</u>	10
22. <u>Vanadium</u>	50
23. <u>Zinc</u>	20
Other:	
Cyanide	10

NR - Not required



## Appendix IV

### Definition and Procedure for the Determination of the Method Detection Limit

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero and determined from analysis of a sample in a given matrix containing analyte.

#### Scope and Application

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device- or instrument-independent.

#### Procedure

1. Make an estimate of the detection limit using one of the following:
  - (a) The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5. If the criteria for qualitative identification of the analyte is based upon pattern recognition techniques, the least abundant signal necessary to achieve identification must be considered in making the estimate.
  - (b) The concentration value that corresponds to three times the standard deviation of replicate instrumental measurements for the analyte in reagent water.
  - (c) The concentration value that corresponds to the region of the standard curve where there is a significant change in sensitivity at low analyte concentrations, i.e., a break in the slope of the standard curve.
  - (d) The concentration value that corresponds to known instrumental limitations.

It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the estimate of the detection limit.
2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.
3. (a) If the MDL is to be determined in reagent water (blank), prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated MDL (Recommend between 1 and 5 times the estimated MDL) Proceed to Step 4.

- (b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated MDL, proceed to Step 4.

If the measured concentration of analyte is less than the estimated MDL, add a known amount of analyte to bring the concentration of analyte to between one and five times the MDL. In the case where an interference is coanalyzed with the analyte:

If the measured level of analyte is greater than five times the estimated MDL, there are two options:

- (1) Obtain another sample of lower level of analyte in same matrix if possible.
  - (2) The sample may be used as is for determining the MDL if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.
4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the MDL and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If blank measurements are required to calculate the measured level of analyte, obtain separate blank measurements for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.
- (b) It may be economically and technically desirable to evaluate the estimated MDL before proceeding with 4a. This will: (1) prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an incorrect MDL can be calculated from data obtained at many times the real MDL even though the background concentration of analyte is less than five times the calculated MDL. To insure that the estimate of the MDL is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower MDL. Take two aliquots of the sample to be used to calculate the MDL and process each through the entire method, including blank measurements as described above in 4a. Evaluate these data:
- (1) If these measurements indicate the sample is in the desirable range for determining the MDL, take five additional aliquots and proceed. Use all seven measurements to calculate the MDL.
  - (2) If these measurements indicate the sample is not in the correct range, reestimate the MDL, obtain new sample as in 3 and repeat either 4a or 4b.

5. Calculate the variance ( $S^2$ ) and standard deviation ( $S$ ) of the replicate measurements, as follows:

$$S^2 = \frac{1}{n-1} \left[ \sum_{i=1}^n X_i^2 - \frac{\left( \sum_{i=1}^n X_i \right)^2}{n} \right]$$

$$S = (S^2)^{1/2}$$

where: the  $x_i$ ,  $i = 1$  to  $n$  are the analytical results in the final method reporting units obtained from the  $n$  sample aliquots and  $\sum_{i=1}^n X_i^2$  refers to the sum of the  $X$  values from  $i = 1$  to  $n$ .

6. (a) Compute the MDL as follows:

$$MDL = 1.645 \cdot 1 - \alpha \cdot S$$

where:

MDL = the method detection

$t_{n-1, 1-\alpha} = .99$  = the student's t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table.

S = standard deviation of the replicate analyses.

- (b) The 95% confidence limits for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution ( $\chi^2/df$ ) and calculated as follows:

$$MDL_{LCL} = 0.69 MDL$$

$$MDL_{UCL} = 1.92 MDL$$

where  $MDL_{LCL}$  and  $MDL_{UCL}$  are the lower and upper 95% confidence limits respectively based on seven aliquots.

7. Optional iterative procedure to verify the reasonableness of the estimated MDL and calculated MDL of subsequent MDL determinations.
- (a) If this is the initial attempt to compute MDL based on the estimated MDL in Step 1, take the MDL as calculated in Step 6, spike in the matrix at the calculated MDL and proceed through the procedure starting with Step 4.
- (b) If the current MDL determination is an iteration of the MDL procedure for which the spiking level does not permit qualitative identification, report the MDL as that concentration between the current spike level and the previous spike level which allows qualitative identification.
- (c) If the current MDL determination is an iteration of the MDL procedure and the spiking level allows qualitative identification, use  $S^2$  from the current MDL calculation and  $S^2$  from the previous MDL calculation to compute the F ratio.

$$\text{if } \frac{S_A^2}{S_B^2} < 3.05$$

then compute the pooled standard deviation by the following equation:

$$S_{\text{pooled}} = \left[ \frac{6S_A^2 + 6S_B^2}{12} \right]^{1/2}$$

if  $\frac{S_A^2}{S_B^2} > 3.05$ , respoke at the last calculated MDL and process the samples through the procedure starting with Step 4.

- (c) Use the  $S_{\text{pooled}}$  as calculated in 7b to compute the final MDL according to the following equation:

$$MDL = 2.681 (S_{\text{pooled}})$$

where 2.681 is equal to  $t_{12, 1-\alpha} = .99$ .

- (d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution.

$$MDL_{LCL} = 0.72 MDL$$

$$MDL_{UCL} = 1.65 MDL$$

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

### Reporting

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units. If the analytical method permits options which affect the method detection limit, these conditions must be specified with the MDL value. The sample matrix used to

determine the MDL must also be identified with the MDL value. Report the mean analyte level with the MDL. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, report the mean recovery, and indicate if the MDL determination was iterated.

If the level of the analyte in the sample-matrix exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL.

### Reference

Glaser, J. A., Foerst, D. L., McKee, G. D., Quave, S. A., and Budde, W. L., "Trace Analysis for Wastewaters," *Environmental Science and Technology*, 15, 1426 (1981).

*Table of Students' t Values at the 99 Percent Confidence Level*

<i>Number of Replicates</i>	<i>Degrees of Freedom (n-1)</i>	<i>t<sub>n-1, 1-α = .99</sub></i>
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
∞	∞	2.326

TABLE 1. SURROGATE SPIKING COMPOUNDS

## SURROGATE RECOVERIES

COMPOUND	FRACTION	WATER		SOIL	
		SPIKE ADDED (µg)	RECOVERY LIMIT (%)	SPIKE ADDED (µg)	RECOVERY LIMIT (%)
Toluene-D8	VOA	50	88-110	50	88-117
4-Bromofluorobenzene	VOA	50	86-115	50	74-121
1,2 Dichloroethane-D4	VOA	50	76-114	50	70-121
Nitrobenzene-D5	BNA	50	35-114	100	23-120
2-Fluorobiphenyl	BNA	50	43-116	100	30-115
p-Terphenyl-D-14	BNA	50	33-141	100	18-137
Phenol-D5	BNA	50	10-94	200	24-113
2-Fluorophenol	BNA	50	21-100	200	25-121
2,4,6-Tribromophenol	BNA	50	10-123	100	19-122
Dibutylchloroendate	Pest.	0.1	24-154	0.1	(20-150)

TABLE 2. BFB KEY ION ABUNDANCE CRITERIA

MASS	ION ABUNDANCE CRITERIA
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 174
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	Greater than 95% but less than 100% of mass 174
177	5 to 9% of mass 176

TABLE 3. DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

MASS	ION ABUNDANCE CRITERIA
51	30-60% of mass 98
68	Less than 2% of mass 69
69	Mass 69 relative abundance
70	Less than 2% of mass 69
127	40-60% of mass 198
197	Less than 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	Greater than 1% of mass 198
441	Present but less than mass 443
442	Greater than 40% of mass 198
443	17-23% of mass 442

TABLE A-1<sup>a</sup>  
LIST OF EPA-APPROVED INORGANIC TEST PROCEDURES

Parameter, units, and method	Reference (method No. or page)				
	EPA 1070	Standard methods 15th Ed.	ASTM	USGS <sup>1</sup>	Other
1. Acidity, as CaCO <sub>3</sub> , mg/L: Electrometric end point or phenolphthalein end point.	305.1	402(4.d)	D1067-70(E)		
2. Alkalinity, as CaCO <sub>3</sub> , mg/L: Electrometric or colorimetric:					
Titration to pH 4.5, manual	310.1	403	D1067(D)	I-1030-78	P. 648. <sup>2</sup>
Or automated	310.2			I-2030-78	
3. Aluminum—Total <sup>3</sup> , mg/L: Digestion <sup>3</sup> followed by:				I-3051-78	
AA direct aspiration	202.1	303C			
AA furnace	202.2	304			Method 200.7. <sup>4</sup>
Inductively coupled plasma					
Or colorimetric (Eriochrome cyanine R)		306B			
4. Ammonia (as N), mg/L: Manual distillation <sup>5</sup> (at pH 9.5):					
Followed by	350.2	417A	D1426-78(A)	I-3520-78	P. 653. <sup>2</sup>
Nesslerization	350.2	417B			
Titration	350.2	417D			
Electrode	350.3		D1426-78(D)		
Automated phenate, or	350.1	417F	D1426-78(C)	I-4523-78	
Automated electrode					
5. Antimony—Total <sup>3</sup> , mg/L: Digestion <sup>3</sup> followed by:					
AA direct aspiration	204.1	303A			
AA furnace, or	204.2	304			Method 200.7. <sup>4</sup>
Inductively coupled plasma					
6. Arsenic—Total <sup>3</sup> , mg/L:					
Digestion <sup>3</sup> followed by	206.5			I-3062-78	
Hydride	206.3	303E	D2972-78(B)		
AA furnace	206.2	304			Method 200.7. <sup>4</sup>
Inductively coupled plasma					
Or, colorimetric (SDDC)	206.4	307B	D2972-78(A)	I-3060-78	
7. Barium—Total <sup>3</sup> , mg/L: Digestion <sup>3</sup> followed by:				I-3064-78	
AA direct aspiration	208.1	303C			
AA furnace, or	208.2	304			Method 200.7. <sup>4</sup>
Inductively coupled plasma					
8. Beryllium—Total <sup>3</sup> , mg/L: Digestion <sup>3</sup> followed by:				I-3006-78	
AA direct aspiration	210.1	303C	D3845-78		
AA furnace	210.2	304			

<sup>a</sup>Table ID in 49FR43251.

TABLE A-1 (Continued)  
LIST OF EPA-APPROVED INORGANIC TEST PROCEDURES

Parameter, units, and method	Reference (method No. or page)				
	EPA 1979	Standard methods 15th Ed.	ASTM	USGS <sup>1</sup>	Other
Inductively coupled plasma Or colorimetric (aluminum).....		309B			Method 200.7. <sup>4</sup>
9. Biochemical oxygen demand (BOD <sub>5</sub> ), mg/L: Winkler (Azide modification).....	405.1	507		I-1578-78	P. 17. <sup>4</sup> P. 548. <sup>4</sup>
Or electrode method.....					
10. Boron—Total, mg/L: Colorimetric (curcumin) or.....	212.3	404A		I-3112-78	Method 200.7. <sup>4</sup>
Inductively coupled plasma.....					P. 844. <sup>4</sup>
11. Bromide, mg/L: Titrimetric.....	320.1		D1246-77(C)	I-1125-78	
12. Cadmium—Total <sup>4</sup> , mg/L: Digestion <sup>4</sup> followed by:					
AA direct aspiration.....	213.2	303A or 303B	D3557-78 (A or B)	I-3135-78 or I-3136-78	Pg. 557. <sup>4</sup>
AA furnace.....	213.2	304			P. 37. <sup>4</sup>
Inductively coupled plasma.....					Method 200.7. <sup>4</sup>
Voltammetry <sup>4</sup> or.....			D3557-78(C)		
Colorimetric (Dithizone).....		310D			
13. Calcium—Total <sup>4</sup> , mg/L: Digestion <sup>4</sup> followed by:					
Atomic absorption.....	215.1	303A	D511-77(C)	I-3152-78	Method 200.7. <sup>4</sup>
Inductively coupled plasma.....					
Or EDTA titration.....	215.2	311C	D511-77(B)		
14. Carbonaceous Biochemical oxygen demand (CBOD <sub>5</sub> ), mg/L: Winkler (Azide modification) or electrode method with nitrification inhibitor.		607(5.e.6)			
15. Chemical oxygen demand (COD), mg/L:					
Titrimetric colorimetric.....	410.1	508A	D1252-78	I-3580-78	P. 550 <sup>4</sup> and
Manual or.....	410.2			I-3582-78	P. 17 <sup>4</sup> and
	410.3			I-3581-78	(1 <sup>4</sup> )
Automated.....	410.4				(1 <sup>4</sup> )
Spectrophotometric.....					
16. Chloride, mg/L:					
Titrimetric (silver nitrate) or.....		407A	D512-67(B)	I-1183-78	
Mercuric nitrate.....	325.3	407B	D512-67(A)	I-1184-78	P. 554. <sup>4</sup>
Colorimetric (ferricyanide) manual or.....			D512-67(C)	I-1187-78	
Automated.....	325.1 or 325.2	407D		I-2187-78	
17. Chlorine—Total residual, mg/L:					
Titrimetric-mercurimetric <sup>4</sup> .....	330.1	408C	D1253-76(A)		
Starch end point.....	330.2	408D			
Iodometric or.....	330.3	408A	D1253-76(B)		
DPD-FAS.....	330.4	408D			
Spectrophotometric, DPD; or.....	330.5	408E			
Electrode.....					(1 <sup>4</sup> )

TABLE A-1 (Continued)  
LIST OF EPA-APPROVED INORGANIC TEST PROCEDURES

Parameter, units, and method	Reference (method No. or page)				
	EPA 1070	Standard methods 16th Ed.	ASTM	USGS <sup>1</sup>	Other
18. Chromium VI dissolved, mg/L: 0.45 micron filtration with:					
Extraction and atomic absorption, or	218.4	303B		I-1232-78	
Colorimetric (Diphenylcarbazide)				I-1230-78	
19. Chromium—Total <sup>2</sup> , mg/L:					
Digestion <sup>3</sup> (optional extraction) followed by	218.3				P. 557. <sup>2</sup>
AA direct aspiration	218.1	303A or 303B	D1687-77(D)	I-3236-78	
AA furnace	218.2	304			Method 200.7. <sup>4</sup>
Inductively coupled plasma					
Or colorimetric (Diphenylcarbazide)		312A	D1687-77(A)		
20. Cobalt—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by:					P. 37. <sup>2</sup>
AA direct aspiration	219.1	303A or 303B	D3558-77 (A or B)	I-3240-78 or I-3238-78	
AA furnace, or	219.2	304			Method 200.7. <sup>4</sup>
Inductively coupled plasma					
21. Color, platinum Cobalt units or dominant wavelength hue, luminance, purity:					( <sup>14</sup> )
Colorimetric, ADMI	110.1	204D			
Platinum cobalt; or	110.2	204A		I-1250-78	
Spectrophotometric	110.3	204B			
22. Copper—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by:					P. 557. <sup>2</sup> and P. 37. <sup>2</sup>
AA direct aspiration	220.1	303A or 303B	D1688-77 (D or E)	I-3271-78 or I-3270-78	
AA furnace	220.2	304			Method 200.7. <sup>4</sup>
Inductively coupled plasma					
Colorimetric (Neocuproine)		313D	D1688-77(A)		( <sup>14</sup> )
Bicinchonate					
23. Cyanide—Total mg/L:					P. 22. <sup>2</sup>
Manual distillation with MgCl <sub>2</sub>	335.2	412D			
Followed by titrimetric	335.2	412B			
Manual or	335.2	412C	D2036-75(A)		
Automated <sup>10</sup> spectrophotometric	335.3	412D	D2036-75(A)	I-3300-78	
	335.1	412F	D2033-75(B)		
24. Cyanide amenable to chlorination, mg/L: Manual distillation with MgCl <sub>2</sub> ; Followed by titrimetric, manual or automated <sup>10</sup> spectrophotometric.					
25. Fluoride—Total, mg/L:					
Manual distillation <sup>9</sup>		413A			
Followed by manual or	340.2	413B	D1170-72(D)	I-4327-78	
Automated electrode					
SPADNS	340.1	413C	D1170-72(A)		
Or automated complexone	340.3	413E			
26. Gold—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by:					
AA direct aspiration	231.1	303A			
Or AA furnace	231.2	304			
27. Hardness—Total as CaCO <sub>3</sub> , mg/L:					
Automated colorimetric	130.1				
EDTA titration	130.2	314B	D1126-67(B)	I-1338-78	Method 200.7. <sup>4</sup>
Inductively coupled plasma					
Or atomic absorption (sum	215.1 †	303A		I-3153-78 †	
of Ca and Mg as their respective carbonates)	242.1			I-3448-78	



TABLE A-1 (Continued)  
LIST OF EPA-APPROVED INORGANIC TEST PROCEDURES

Parameter, units, and method	Reference (method No. or page)				
	EPA 1070	Standard methods 15th Ed.	ASTM	USGS <sup>1</sup>	Other
29. Hydrogen ion (pH), pH units: Electrometric .....	150.1 .....	423 .....	D1203-76(A) or D1203-76(B) .....	I-1586-78 .....	(10)
Measurements; or automated electrode .....					
29. Iridium—Total <sup>2</sup> , mg/L: Digestion <sup>2</sup> followed by: AA direct aspiration .....	235.1 .....	303A .....			P. 557. <sup>2</sup>
Or AA furnace .....	235.2 .....	304 .....			
30. Iron—Total <sup>2</sup> , mg/L: Digestion <sup>2</sup> followed by .....		303A or 303B .....	D1068-77 .....	I-3381-78 .....	Method 200.7. <sup>2</sup>
AA direct aspiration .....	236.1 .....	303B .....	(C or D) .....		
AA furnace .....	236.2 .....	304 .....			(11)
Inductively coupled plasma .....					
Or colorimetric (Phenanthroline) .....		315B .....	D1068-77(A) .....		P. 552. <sup>2</sup>
31. Kjeldahl nitrogen—Total (as N), mg/L: Digestion and distillation .....	351.3 .....	420A or B .....			
Followed by titration .....	351.3 .....	417D .....	D3500-77 .....		P. 557. <sup>2</sup>
Nesslerization or .....	351.3 .....	417B .....			
Electrode .....	351.3 .....	417E .....		I-4551-78 .....	Method 200.7. <sup>2</sup>
Automated phenate .....	351.1 .....			I-4552-78 .....	
Semi-automated block digester .....	351.2 .....				P. 557. <sup>2</sup>
Or potentiometric .....	351.4 .....				
32. Lead—Total <sup>2</sup> , mg/L: Digestion <sup>2</sup> followed by: AA direct aspiration .....	239.1 .....	303A or 303B .....	D3559-78 (A or B) .....	I-3389-78 .....	Method 200.7. <sup>2</sup>
AA furnace .....	239.2 .....	304 .....			
Inductively coupled plasma .....					P. 557. <sup>2</sup>
Voltametry <sup>2</sup> or .....			D3559-78(C) .....		
Colorimetric (Dithizone) .....		316B .....			Method 200.7. <sup>2</sup>
33. Magnesium—Total <sup>2</sup> , mg/L: Digestion <sup>2</sup> followed by: Atomic absorption .....	242.1 .....	303A .....	D511-77(B) .....	I-3447-78 .....	
Inductively coupled plasma .....					P. 557. <sup>2</sup>
Or gravimetric .....		316B .....	D511-77(A) .....		
34. Manganese—Total <sup>2</sup> , mg/L: Digestion <sup>2</sup> followed by: AA direct aspiration .....	243.1 .....	303A or 303B .....	D858-77 (B or C) .....	I-3454-78 .....	Method 200.7. <sup>2</sup>
AA furnace .....	243.2 .....	304 .....			
Inductively coupled plasma .....					P. 564. <sup>2</sup>
Or colorimetric (Periodate) .....		316B .....	D858-77(A) .....		
Periodate .....					18.

TABLE A-1 (Continued)  
LIST OF EPA-APPROVED INORGANIC TEST PROCEDURES

Parameter, units, and method	Reference (method No. or page)				
	EPA 1979	Standard methods 16th Ed.	ASTM	USQ8 <sup>1</sup>	Other
35. Mercury—Total <sup>2</sup> , mg/L: Cold vapor, manual or _____ Automated _____	245.1 _____ 245.2 _____	303F _____	D3223-78 _____	I-3482-78 _____	P. 559. <sup>2</sup>
36. Molybdenum—Total <sup>2</sup> , mg/L: Digestion <sup>2</sup> followed by: AA direct aspiration _____ AA furnace, or _____ Inductively coupled plasma _____	248.1 _____ 248.2 _____	303C _____ 304 _____		I-3490-78 _____	Method 200.7. <sup>2</sup>
37. Nickel—Total <sup>2</sup> , mg/L: Digestion <sup>2</sup> followed by: AA direct aspiration _____ AA furnace _____ Inductively coupled plasma _____ Or colorimetric (1-leptoxime) _____	249.1 _____ 249.2 _____	303A or 303B _____ 304 _____ 321D _____	D1886-77 (C or D) _____	I-3498-78 _____	Method 200.7. <sup>2</sup>
38. Nitrate (as N), mg/L: Bicinch sulfate, or _____ Nitrate-nitrite N minus Nitrite N _____	352.1 _____ See parameters 39 and 40.	See parameters 39 and 40.	D002-71 _____ See parameters 39 and 40.	See parameters 39 and 40.	P. 554. <sup>2</sup> P. 28. <sup>2</sup>
39. Nitrate-nitrite (as N), mg/L: Cadmium reduction, manual _____ Or automated; or _____ Automated hydrazine _____	353.3 _____ 353.2 _____ 353.1 _____	418C _____ 418F _____	D3867-79(B) _____ D3867-79(A) _____	I-4545-78 _____	
40. Nitrite (as N), mg/L: Spectrophotometric, manual or _____ Automated (Diazotization) _____	354.1 _____	419 _____	D1254-87 _____	I-4540-78 _____	18.
41. Oil and grease—Total recoverable, mg/L: Gravimetric (extraction).	413.1 _____	503A _____			P. 551 <sup>2</sup> and P. 4. <sup>22</sup>
42. Organic carbon—Total (TOC), mg/L: Combustion or oxidation.	415.1 _____	505 _____	D2579-78(A) or D2579-78(B).		PP. 552-53. <sup>2</sup>
43. Organic nitrogen (as N), mg/L: Total Kjeldahl N minus ammonia N.	See parameters 31 and 4.	420A _____	D3590-77 minus D1426-70(A).	See parameters 31 and 4.	P. 561. <sup>2</sup>
44. Orthophosphate (as P), mg/L: Ascorbic acid method, automated Or manual single reagent or _____ Manual two reagent _____	365.1 _____ 365.2 _____ 365.3 _____	424G _____ 424F _____	D515-78(A) _____	I-4601-78 _____	
45. Oxmium—Total <sup>2</sup> , mg/L: Digestion <sup>2</sup> followed by: AA direct aspiration, or _____ AA furnace _____	252.1 _____ 252.2 _____	303C _____ 304 _____			
46. Oxygen, dissolved, mg/L: Winkler (Azide modification) _____ Or electrode _____	360.2 _____ 360.1 _____	421B _____ 421F _____	D1589-80(A) _____	I-1575-78 _____ I-1576-78 _____	P. 550. <sup>2</sup>
47. Palladium—Total <sup>2</sup> , mg/L: Digestion <sup>2</sup> followed by: AA direct aspiration _____ Or AA furnace _____	253.1 _____ 253.2 _____				P. 527. <sup>22</sup> P. 528. <sup>22</sup>
48. Phenols, mg/L: Manual distillation _____ Followed by manual _____ Or automated <sup>12</sup> colorimetric (4AAP) _____	420.1 _____ 420.1 _____ 420.2 _____		D1783-70 (A or B) _____		20. 20. 21.
49. Phosphorus (solvent-free), mg/L: (See Bould chro-					

TABLE A-1 (Continued)  
LIST OF EPA-APPROVED INORGANIC TEST PROCEDURES

Parameter, units, and method	Reference (method No. or page)				
	EPA 1070	Standard methods 15th Ed.	ASTM	USGS <sup>1</sup>	Other
50. Phosphorus—Total, mg/L: Persulfate digestion.....	365.2	424C (III).....			P. 561. <sup>2</sup>
Followed by manual or .....	365.2 or 365.3	424F.....	D515-76(A).....		
Automated ascorbic acid.....	365.1	424G.....		I-1600-78 .....	
Reduction; or semi-automated block digester.....	365.4			I-1603-78 .....	
51. Platinum—Total <sup>3</sup> , mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration.....	255.1	303A.....			P. 560. <sup>2</sup> Method 200.7. <sup>4</sup>
Or AA furnace.....	255.2	304.....			
52. Potassium—Total <sup>3</sup> , mg/L: Digestion <sup>3</sup> followed by: Atomic absorption.....	250.1	303A.....		I-3030-78 .....	
Inductively coupled plasma.....					
Or flame photometric.....		322B.....	D1428-64(A).....		
53. Residue—total, mg/L: Gravimetric, 103-105°C.....	160.3	209A.....		I-3750-78 .....	
54. Residue—filterable, mg/L: Gravimetric, 180°C.....	160.1	209B.....		I-1750-78 .....	
55. Residue—nonfilterable, (TSS), mg/L: Gravimetric, 103-105°C post washing of residue.	160.2	209D.....		I-3765-78 .....	
56. Residue—settlesable, mg/L: Volumetric (Imhoff cone) or gravimetric.	160.5	209F.....			
57. Residue—volatile, mg/L: Gravimetric, 550°C.....	160.4	209E.....		I-3753-78 .....	
58. Rhodium—Total <sup>3</sup> , mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration.....	265.1	303A.....			Method 200.7. <sup>4</sup>
Or AA furnace.....	267.2	304.....			
59. Ruthenium—Total <sup>3</sup> , mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration.....	267.1	303A.....			
Or AA furnace.....	267.2	304.....			
60. Selenium—Total <sup>3</sup> mg/L: Digestion <sup>3</sup> followed by: AA furnace.....	270.2	304.....			
Inductively coupled plasma.....					
Or hydride.....	270.3	303E.....	D3859-78 .....	I-3667-78 .....	
61. Silica—Dissolved, mg/L: 0.45 micron filtration: Followed by manual or .....	370.1	425C.....	D659-68(B).....	I-1700-78 .....	
Automated colorimetric (Molybdoasulfate), or .....				I-2700-78 .....	
Inductively coupled plasma.....					Method 200.7. <sup>4</sup>
62. Silver—Total <sup>3</sup> mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration.....	272.1	303A or 303B.....		I-3720-78 .....	P. 557 <sup>2</sup> and p. 37. <sup>2</sup>
AA furnace, or .....	272.1	304.....			
Inductively coupled plasma.....					Method 200.7. <sup>4</sup>

TABLE A-1 (Continued)  
LIST OF EPA-APPROVED INORGANIC TEST PROCEDURES

Parameter, units, and method	Reference (method No. or page)				
	EPA 1070	Standard methods 18th Ed.	ASTM	USGS <sup>1</sup>	Other
63. Sodium—Total <sup>a</sup> , mg/L: Digestion <sup>a</sup> followed by: Atomic absorption.....	273.1.....	303A.....		I-3735-78.....	P. 561. <sup>a</sup> Method 200.7. <sup>a</sup>
Inductively coupled plasma.....			D1428-84(A).....		
Or flame photometric.....			D1125-77(A).....	I-1780-78.....	P. 547. <sup>a</sup>
64. Specific conductance, mhos/cm: Wheatstone bridge.....	120.1.....	205.....			
65. Sulfate (as SO <sub>4</sub> ), mg/L: Automated methylthymol blue.....	376.2.....			I-2822-78.....	PP. 562-63. <sup>a</sup>
Gravimetric, or.....	375.3.....	426A or 426B.....	D516-66(A).....		
Turbidimetric.....	376.4.....	426C.....	D516-66(B).....		
66. Sulfide (as S), mg/L: Titrimetric (iodine) or.....	376.1.....	427D.....		I-3840-78.....	
Colorimetric (methylene blue).....	376.2.....	427C.....			
67. Sulfite (as SO <sub>3</sub> ), mg/L: Titrimetric (iodine iodate).....	377.1.....	426F.....	D1339-78(C).....		
68. Surfactants, mg/L: Colorimetric (methylene blue).....	425.1.....	612A.....	D2330-66(A).....		( <sup>a</sup> )
69. Temperature, °C: Thermometric.....	170.1.....	212.....			
70. Thallium—Total <sup>a</sup> , mg/L: Digestion <sup>a</sup> followed by: AA direct aspiration.....	279.1.....	303A.....			
AA furnace, or.....	279.2.....	304.....			Method 200.7. <sup>a</sup>
Inductively coupled plasma.....					
71. Tin—Total <sup>a</sup> , mg/L: Digestion <sup>a</sup> followed by: AA direct aspiration or.....	282.1.....	303A.....		I-3850-78.....	
AA furnace.....	282.2.....	304.....			
72. Titanium—Total <sup>a</sup> , mg/L: Digestion <sup>a</sup> followed by: AA direct aspiration or.....	283.1.....	303C.....			
AA furnace.....	283.2.....	304.....			
73. Turbidity, NTU: Nephelometric.....	180.1.....	214A.....	D1889-71.....	I-3860-78.....	
74. Vanadium—Total <sup>a</sup> , mg/L: Digestion <sup>a</sup> followed by: AA direct aspiration.....	286.1.....	303C.....			
AA furnace.....	286.2.....	304.....			Method 200.7. <sup>a</sup>
Inductively coupled plasma.....			D3373-76.....		
Or colorimetric (Gallio Eckf).....					
75. Zinc—Total <sup>a</sup> , mg/L: Digestion <sup>a</sup> followed by: AA direct aspiration.....	289.1.....	303A or 303B.....	D1691-77(D).....	I-3000-78.....	P. 557. <sup>a</sup> P. 37. <sup>a</sup>
AA furnace.....	289.2.....	304.....	D1691-77(C).....		Method 200.7. <sup>a</sup>
Inductively coupled plasma.....					<sup>a</sup> .
Or colorimetric (Zincon).....					

TABLE A-1 (Concluded)  
LIST OF EPA-APPROVED INORGANIC TEST PROCEDURES

Table Notes

<sup>1</sup> "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments," U.S. Department of the Interior, U.S. Geological Survey, Open-File Report 78-879, or "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments," N.W. Skougstad, *et al*, U.S. Geological Survey, Techniques of Water-Resources Investigation, Book 5, Chapter A1, 1979.

<sup>2</sup> "Official Methods of Analysis of the Association of Official Analytical Chemists" methods manual, 13th ed. (1980).

<sup>3</sup> For the determination of total metals the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic-metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes, 1979." One (§ 4.1.3), is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrochloric acids (§ 4.1.4) is preferred; however, the analyst should be cautioned that this mild digestion may not suffice for all sample types. Particularly, if a colorimetric procedure is to be employed, it is necessary to ensure that all organo-metallic bonds be broken so that the metal is in a reactive state. In those situations, the vigorous digestion is to be preferred making certain that at no time does the sample go to dryness. Samples containing large amounts of organic materials would also benefit by this vigorous digestion. Use of the graphite furnace technique, inductively coupled plasma, as well as determinations for certain elements such as arsenic, the noble metals, mercury, selenium, and titanium require a modified digestion and in all cases the method write-up should be consulted for specific instructions and/or cautions.

Note: If the digestion procedure for direct aspiration or graphite furnace atomic absorption analysis included in one of the other approved references is different than the above, the EPA procedure must be used.

Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane filter. Following filtration of the sample, the referenced procedure for total metals must be followed. Sample digestion of the filtrate for dissolved metals, or digestion of the original sample solution for total metals may be omitted for AA (direct aspiration or graphite furnace) and ICP analyses provided the sample has a low COD and the filtrate meets the following criteria:

- (a) is visibly transparent
- (b) has no perceptible odor, and
- (c) is free of particulate or suspended matter following acidification.

<sup>4</sup> The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes," is given at Appendix C of this Part 130.

<sup>5</sup> Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.

<sup>6</sup> Ammonia, Automated Electrode Method, Industrial Method Number 379-75WE, dated February 10, 1976, Technicon AutoAnalyzer II, Technicon Industrial Systems, Tarrytown, New York 10591.

<sup>7</sup> Carbonaceous biochemical oxygen demand (CBOD<sub>5</sub>) must not be confused with the traditional BOD<sub>5</sub> test which measures "total BOD". The addition of the nitrification inhibitor is not a procedural option, but must be included to report the CBOD<sub>5</sub> parameter. A discharger whose permit requires reporting the traditional CBOD<sub>5</sub> may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states CBOD<sub>5</sub> is required can the permittee report data obtained using the nitrification inhibitor.

<sup>8</sup> American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, NY 10018.

<sup>9</sup> The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.

<sup>10</sup> Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

<sup>11</sup> COD Method, Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, Texas 77840.

<sup>12</sup> The back titration method will be used to resolve controversy.

<sup>13</sup> National Council of the Paper Industry for Air and Stream Improvement, Inc., Technical Bulletin 253, December 1971.

<sup>14</sup> Copper, Bichromate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

<sup>15</sup> After the manual distillation is completed, the auto-analyzer manifolds in EPA Methods 335.03 (Cyanide) or 420.2 (phenols) are simplified by connecting the re-sample line directly to the sampler. When using the manifold setup shown in Method 335, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2.

<sup>16</sup> Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 376-75WA, October 1976, Technicon Auto-Analyzer II, Technicon Industrial Systems, Tarrytown, New York 10591.

<sup>17</sup> Iron, 1,10-Phenanthroline Method, Method 8006, 1980, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

<sup>18</sup> Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2-113 and 2-117, Hach Chemical Company, Loveland, Colorado 80537.

<sup>19</sup> Nitrogen, Nitrite, Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

<sup>20</sup> Gmelin, D., Brown, E., "Methods for Analysis of Organic Substances in Water," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A3, p.4 (1972).

<sup>21</sup> R.F. Addison and R.G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," Journal of Chromatography, Vol. 47, No. 3, pp. 421-426, 1970.

<sup>22</sup> Recommended methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to a pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the recommended method is satisfactory.

<sup>23</sup> Stevens, H.H., Ficke, J.F., and Smoot, G.F., "Water Temperature-Influential Factors, Field Measurement and Data Presentation," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 1, Chapter D1, 1975.

<sup>24</sup> Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2-231 and 2-333, Hach Chemical Company, Loveland, Colorado 80537.

<sup>25</sup> "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater* (1981).

<sup>26</sup> The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater*, 14th Edition. The colorimetric reaction is conducted at a pH of 10.0 ± 0.2. The approved methods are given on pp. 576-81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrophotometric procedure.

<sup>27</sup> ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977, Orion Research Incorporated, 840 Memorial Drive, Cambridge, Massachusetts 02138.

# APPENDIX VI

TABLE A-2  
LIST OF EPA-APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter <sup>1</sup>	EPA Method Number <sup>2</sup>			Other
	GC	GC/MS	IM/LO	
1. Acenaphthene.....	810	825, 1825	810	Note 2, p. 11;
2. Acenaphthylene.....	810	825, 1825	810	
3. Acridin.....	803	824, 1824		
4. Acrylonitrile.....	803	824, 1824		Note 2, p. 11;
5. Anthracene.....	810	825, 1825	810	
6. Benzene.....	802	824, 1824		
7. Benztidine.....		825, 1825	805	Note 2, p. 11;
8. Benzofuran.....	810	825, 1825	810	
9. Benzofuran.....	810	825, 1825	810	
10. Benzofuran.....	810	825, 1825	810	Note 2, p. 11;
11. Benzofuran.....	810	825, 1825	810	
12. Benzofuran.....	810	825, 1825	810	
13. Benzyl Chloride.....				Note 2, p. 11;
14. Benzyl Chloride.....				
15. Benzyl Chloride.....				
16. Bis(2-chloroethyl) methane.....	806	825, 1825		Note 2, p. 11;
17. Bis(2-chloroethyl) ether.....	811	825, 1825		
18. Bis(2-ethylhexyl) phthalate.....	806	825, 1825		
19. Bromochloromethane.....	801	824, 1824		Note 2, p. 11;
20. Bromoform.....	801	824, 1824		
21. Bromomethane.....	801	824, 1824		
22. 4-Bromophenyl phenyl ether.....	811	825, 1825		Note 2, p. 11;
23. Carbon tetrachloride.....	801	824, 1824		
24. Chlorobenzene.....	801	824, 1824		
25. Chloroethane.....	801	824, 1824		Note 2, p. 11;
26. 2-Chloroethyl methyl ether.....	801	824, 1824		
27. Chloroform.....	801	824, 1824		
28. Chloromethane.....	812	825, 1825		Note 2, p. 11;
29. 2-Chloronaphthalene.....	801	824, 1824		
30. 2-Chlorophenol.....	801	824, 1824		
31. 4-Chlorophenyl phenyl ether.....	811	825, 1825		Note 2, p. 11;
32. Chloroethane.....	810	825, 1825	810	
33. Chloroethane.....	810	825, 1825	810	
34. Chloroethane.....	801	824, 1824		Note 2, p. 11;
35. 1,2-Dichlorobenzene.....	801	824, 1824		
36. 1,3-Dichlorobenzene.....	801	824, 1824		
37. 1,4-Dichlorobenzene.....	801	824, 1824		Note 2, p. 11;
38. 2,3-Dichlorobenzene.....	801	824, 1824		
39. 2,4-Dichlorobenzene.....	801	824, 1824		
40. 1,1-Dichloroethane.....	801	824, 1824		Note 2, p. 11;
41. 1,2-Dichloroethane.....	801	824, 1824		
42. 1,1-Dichloroethane.....	801	824, 1824		
43. 1,1,2,2-Tetrachloroethane.....	801	824, 1824		Note 2, p. 11;
44. 2,4-Dichlorophenol.....	801	824, 1824		
45. 1,2-Dichloropropane.....	801	824, 1824		
46. 1,3-Dichloropropane.....	801	824, 1824		Note 2, p. 11;
47. 1,3-Dichloropropane.....	801	824, 1824		
48. 1,3-Dichloropropane.....	801	824, 1824		
49. 2,4-Dichlorophenol.....	801	824, 1824		Note 2, p. 11;
50. Dimethyl phthalate.....	801	824, 1824		
51. Di-n-butyl phthalate.....	801	824, 1824		
52. Di-n-octyl phthalate.....	801	824, 1824		Note 2, p. 11;
53. 2,4-Dinitrophenol.....	801	824, 1824		
54. 2,4-Dinitrophenol.....	801	824, 1824		
55. 2,6-Dinitrophenol.....	801	824, 1824		Note 2, p. 11;
56. Epichlorohydrin.....	801	824, 1824		
57. Epichlorohydrin.....	801	824, 1824		
58. Epichlorohydrin.....	801	824, 1824		Note 2, p. 11;
59. Epichlorohydrin.....	801	824, 1824		
60. Epichlorohydrin.....	801	824, 1824		

TABLE A-2 (Concluded)  
LIST OF EPA-APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter <sup>1</sup>	EPA Method Number <sup>2,3</sup>			Other
	GC	GC/MS	HPLO	
97. Ethylbenzene	602	624, 1024		
98. Fluorene	610	625, 1025	610	
99. Fluorene	610	625, 1025	610	
100. Hexachlorobenzene	612	625, 1025		
101. Hexachlorobenzene	612	625, 1025		
102. Hexachlorocyclopentadiene	612	625, 1025		
103. Hexachloroethane	612	625, 1025		
104. Methyl 1,3-cyclopentadiene	610	625, 1025	610	
105. Isophorone	606	625, 1025		
106. Methylene Chloride	601	624, 1024		Note 2, p. 130;
107. 2 Methyl-4,6-Dichlorophenol	604	625, 1025		
108. Naphthalene	610	625, 1025		
109. Naphthalene	608	625, 1025		
110. 2 Nitrophenol	604	625, 1025		
111. 4 Nitrophenol	604	625, 1025		
112. Nitroacetylmethylamine	607	625, 1025		
113. Nitroacetyl-n-propylamine	607	625, 1025		
114. Nitroacetylphenylamine	607	625, 1025		
115. 2,2-dimethyl-1-chloropropane	611	625, 1025		
116. PCB-1018	608	625		Note 2, p. 43;
117. PCB-1221	608	625		Note 2, p. 43;
118. PCB-1232	608	625		Note 2, p. 43;
119. PCB-1242	608	625		Note 2, p. 43;
120. PCB-1248	608	625		Note 2, p. 43;
121. PCB-1254	608	625		Note 2, p. 43;
122. PCB-1260	608	625		Note 2, p. 43;
123. Perchlorophenol	604	625, 1025		Note 2, p. 140;
124. Phenanthrene	610	625, 1025	610	
125. Phenol	610	625, 1025		
126. Pyrene	601	624, 1024		Note 2, p. 130;
127. 2,3,7,8-Tetrachlorodibenzo-p-dioxin	601	624, 1024		Note 2, p. 130;
128. 1,1,2,2-Tetrachloroethane	602	624, 1024		Note 2, p. 130;
129. Toluene	612	625, 1025		Note 2, p. 130;
130. 1,2,4-Trichlorobenzene	601	624, 1024		Note 2, p. 130;
131. 1,1-Trichloroethane	601	624, 1024		Note 2, p. 130;
132. 1,1,2-Trichloroethane	601	624, 1024		Note 2, p. 130;
133. Trichloroethane	601	624, 1024		
134. Trichloroacetylmethylamine	601	625, 1025		
135. 2,4,6-Trichlorophenol	601	624, 1024		
136. Vinyl Chloride	601	624, 1024		

Table Note

<sup>1</sup>All parameters are expressed in micrograms per liter (µg/L).

<sup>2</sup>The full text of Methods 601-613, 625, 1024, and 1025, are given in Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given in Appendix B, "Determination of the Method Detection Limit," of this Part 136.

<sup>3</sup>Methods for Benzene, Chlorinated Organic Compounds, Perchlorophenol and Pesticides in Water and Wastewater, U.S. Environmental Protection Agency, September, 1978. Method 624 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds is Method 603 or Method 1074.

<sup>4</sup>Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1025, are preferred methods for these compounds.

<sup>5</sup>625. Screening only.

<sup>6</sup>Subsisted Analytical Methods Approved and Cited by the United States Environmental Protection Agency, Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).

<sup>7</sup>Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 601-613, 624, 625, 1024, and 1025 (See Appendix A of this Part 136) in accordance with procedures each in section 8.2 of each of these Methods. Additionally, each laboratory, on an ongoing basis must make and analyze 10% (1%) of Methods 625 and 625 and 100% for Methods 1074, and 1075) of all samples to monitor and evaluate laboratory data quality in accordance with sections 9.3 and 9.4 of these Methods. When the laboratory is unable to meet the requirements of these Methods, the laboratory must be notified by the EPA.

TABLE A-3<sup>\*</sup>  
LIST OF EPA-APPROVED TEST PROCEDURES FOR PESTICIDE

Parameter (pg/L)	Method	EPA <sup>a,1</sup>	Standard Methods 15th Ed	ASTM	Other
1. Aldrin	GC	608	608A	D3088	Note 3, p. 7; Note 4, p. 30.
	GC/MS	625			
2. Atrazine	GC				Note 3, p. 83; Note 6, p. 548.
3. Atrazine	TLC				Note 3, p. 84; Note 6, p. 548.
4. Atrazine	GC				Note 3, p. 83; Note 6, p. 548.
5. Atrazine	GC				Note 3, p. 83; Note 6, p. 548.
6. Azinphos methyl	GC				Note 3, p. 25; Note 6, p. 551.
7. Barban	TLC				Note 3, p. 104; Note 6, p. 544.
8. $\alpha$ -BHC	GC	608	608A	D3088	Note 3, p. 7.
	GC/MS	625			
9. $\beta$ -BHC	GC	608		D3088	
	GC/MS	625			
10. $\delta$ -BHC	GC	608		D3088	
	GC/MS	625			
11. $\gamma$ -BHC (Lindane)	GC	608	608A	D3088	Note 3, p. 7; Note 4, p. 30.
	GC/MS	625			
12. Captaf	GC		608A		Note 3, p. 7.
13. Carbiaryl	TLC				Note 3, p. 84; Note 6, p. 548.
14. Carboxendosulfon	GC				Note 4, p. 30; Note 6, p. 573.
15. Chlordane	GC	608	608A	D3088	Note 3, p. 7.
	GC/MS	625			
16. Chlorpropham	TLC				Note 3, p. 104; Note 6, p. 544.
17. 2,4-D	GC		608B		Note 3, p. 115; Note 4, p. 38.
18. 4,4'-DDD	GC	608	608A	D3088	Note 3, p. 7; Note 4, p. 30.
	GC/MS	625			
19. 4,4'-DDE	GC	608	608A	D3088	Note 3, p. 7; Note 4, p. 30.
	GC/MS	625			
20. 4,4'-DDT	GC	608	608A	D3088	Note 3, p. 7; Note 4, p. 30.
	GC/MS	625			
21. Demeton-O	GC				Note 3, p. 25; Note 6, p. 551.
22. Demeton-S	GC				Note 3, p. 25; Note 6, p. 551.
23. Dazinon	GC				Note 3, p. 25; Note 4, p. 30; Note 6, p. 551.
24. Dicamba	GC				Note 3, p. 115.
25. Dichlorofenol	GC				Note 4, p. 30; Note 6, p. 573.
26. Dichloran	GC		608A		Note 3, p. 7.
27. Dicolol	GC			D3088	
28. Dieldrin	GC	608	608A		Note 3, p. 7; Note 4, p. 30.
	GC/MS	625			
29. Dioxathion	GC				Note 4, p. 30; Note 6, p. 573.
30. Disulfoton	GC				Note 3, p. ; Note 6, p. 551.
31. Dursin	TLC				Note 3, p. 104; Note 6, p. 544.
32. Endosulfan I	GC	608	608A	D3088	Note 3, p. 7.
	GC/MS	625			
33. Endosulfan II	GC	608	608A	D3088	Note 3, p. 7.
	GC/MS	625			
34. Endosulfan sulfate	GC	608			
	GC/MS	625			
35. Endrin	GC	608	608A	D3088	Note 3, p. 7; Note 4, p. 30.
	GC/MS	625			
36. Endrin alkoxide	GC	608			
	GC/MS	625			
37. Edion	GC				Note 4, p. 30; Note 6, p. 573.
38. Fenitrothion	TLC				Note 3, p. 104; Note 6, p. 544.
39. Fenitrothion-TCA	TLC				Note 3, p. 104; Note 6, p. 544.



TABLE A-3 (Concluded)  
LIST OF EPA-APPROVED TEST PROCEDURES FOR PESTICIDE

Parameter (µg/L)	Method	EPA <sup>a</sup>	Standard Methods 15th Ed	ASTM	Other
40. Heptachlor.....	GC.....	608	608A	D3088	Note 3, p. 7; Note 4, p. 30.
41. Heptachlor epoxide.....	GC/MS.....	625			
	GC.....	608	608A	D3088	Note 3, p. 7; Note 4, p. 30; Note 6, p. 873.
42. Isodrin.....	GC/MS.....	625			
	GC.....				Note 4, p. 30; Note 6, p. 873.
43. Linuron.....	TLC.....				Note 3, p. 104; Note 6, p. 864.
44. Malathion.....	GC.....		608A		Note 3, p. 25; Note 4, p. 30; Note 6, p. 851.
45. Methiocarb.....	TLC.....				Note 3, p. 84; Note 6, p. 860.
46. Methoxychlor.....	GC.....		608A	D3088	Note 3, p. 7; Note 4, p. 30.
47. Monocarbale.....	TLC.....				Note 3, p. 84; Note 6, p. 860.
48. Moxa.....	GC.....		608A		Note 3, p. 7.
49. Monuron.....	TLC.....				Note 3, p. 104; Note 6, p. 864.
50. Monuron-TCA.....	TLC.....				Note 3, p. 104; Note 6, p. 864.
51. Naluron.....	TLC.....				Note 3, p. 104; Note 6, p. 864.
52. Parathion methyl.....	GC.....		608A		Note 3, p. 25; Note 4, p. 30.
53. Parathion ethyl.....	GC.....		608A		Note 3, p. 25.
54. PCNB.....	GC.....		608A		Note 3, p. 7.
55. Perthane.....	GC.....			D3088	
56. Prometon.....	GC.....				Note 3, p. 83; Note 6, p. 868.
57. Prometryn.....	GC.....				Note 3, p. 83; Note 6, p. 868.
58. Propazine.....	GC.....				Note 3, p. 83; Note 6, p. 868.
59. Propham.....	TLC.....				Note 3, p. 104; Note 6, p. 864.
60. Propoxur.....	TLC.....				Note 3, p. 84; Note 6, p. 860.
61. Secbumeton.....	TLC.....				Note 3, p. 83; Note 6, p. 868.
62. Sekuron.....	TLC.....				Note 3, p. 104; Note 6, p. 864.
63. Simazine.....	GC.....				Note 3, p. 83; Note 6, p. 868.
64. Strobane.....	GC.....		608A		Note 3, p. 7.
65. Suxp.....	TLC.....				Note 3, p. 104; Note 6, p. 864.
66. 2,4,5-T.....	GC.....		608B		Note 3, p. 115; Note 4, p. 35.
67. 2,4,5-TP (Saves).....	GC.....		608B		Note 3, p. 115.
68. Terbutylazine.....	GC.....				Note 3, p. 83; Note 6, p. 868.
69. Tonaphene.....	GC.....	608	608A	D3088	Note 3, p. 7; Note 4, p. 30.
	GC/MS.....	625			
70. Trifluralin.....	GC.....		608A		Note 3, p. 7.

Table Notes

- <sup>a</sup> Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table IC, where entries are listed by chemical name.
- <sup>b</sup> The full text of methods 608 and 625 are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 138. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this Part 138.
- <sup>c</sup> Methods for Benzene, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978. This EPA publication includes thin-layer chromatography (TLC) methods.
- <sup>d</sup> "Methods for Analysis of Organic Substances in Water," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A3 (1972).
- <sup>e</sup> The method may be extended to include a-BHC, δ-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.
- <sup>f</sup> "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater* (1981).
- <sup>g</sup> Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A of this Part 138) in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance.
- Note.—These warning limits are promulgated as an "interim final action with a request for comments."

TABLE A-4<sup>a</sup>  
LIST OF EPA-APPROVED RADIOLOGICAL TEST PROCEDURES

Parameter and units	Methods	EPA <sup>b</sup>	Reference (method No. or page)		
			Standard Methods 15th Ed.	ASTM	USGS <sup>c</sup>
Alpha-Total, p <sup>23</sup> per liter .....	Proportional or scintillation counter .....	900.0 .....	703	D1943-66	pp. 75 and 78. <sup>e</sup>
Alpha-Counting error, p <sup>23</sup> per liter .....	Proportional or scintillation counter .....	Appendix B .....	703	D1943-66	p. 78.
Alpha-Counting error, p <sup>23</sup> per liter .....	Proportional counter .....	900.0 .....	703	D1800-66	pp. 75 and 78. <sup>e</sup>
Beta-Counting error, p <sup>23</sup> per liter .....	Proportional counter .....	Appendix B .....	703	D1890-66	p. 78.
(a) Radium-Total, p <sup>226</sup> per liter .....	Proportional counter .....	903.0 .....	705	D2460-70	
(b) <sup>226</sup> Ra, p <sup>226</sup> per liter .....	Scintillation counter .....	903.1 .....	706	D3454-78	p. 81.

Table Notes

- <sup>a</sup> "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-032 (1980 update), U.S. Environmental Protection Agency, August 1980.  
<sup>b</sup> Fishman, M.J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open-File Report 78-177 (1978).  
<sup>c</sup> The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the "total."

<sup>a</sup> Table 1E in 49FR43258.



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**APPENDIX C-3**

**LABORATORY QUALITY ASSURANCE  
QUALITY CONTROL DATA PACKAGE OUTLINE**

In order to ensure the validity of the reported analytical results, the status of the following criteria which determine the quality of the analytical data need to be ascertained:

1. Stability of the sample(s) analyzed.
2. Performance of the instrument(s) used for analyses.
3. Possibility of sample contamination.
4. Identification and quantification of the analyte(s) in the sample(s) analyzed.
5. Precision in analyses.
6. Accuracy of the results reported.

Documents required to establish the status of the above criteria during sample analysis are summarized in the following sections:

1. Stability of Sample(s) Analyzed

To establish the stability of the environmental samples analyzed the lab will provide the following:

- 1(a). Chain-of-custody paper for each sample received.
- 1(b). Date and time of both extraction and analysis of each sample.

2. Performance of the Instrument(s) Used for Analysis

Analytical methodology for analyzing the samples will determine the type of the instrument(s) to be used by the lab. To demonstrate the working condition of the instrument(s) during analyses the lab will submit the following:

- 2(a). Detection limits for all the HSL compounds analyzed.
- 2(b). For GC/MS analysis, a final tune mass spectrum and the quantitation report for the GC/MS tuning compound on each day prior to any analysis.
- 2(c). Data for the initial and continuous calibration of the instrument including the response factor (or calibration factor) for each compound to be analyzed. (The instrument will be calibrated initially using standard solutions, as specified in the protocol, and the initial calibration will be verified on each day prior to sample analysis.)
- 2(d). Raw data (i.e., Data System print-outs or integration reports) for all standard solutions analyzed in both initial calibration and continuous (i.e., daily on-going) calibration.
- 2(e). Identification of each instrument used for analyses.

### 3. Possibilities of Sample Contamination

Each day prior to any analysis the lab will analyze the 'Method Blank' and submit the following:

- 3(a). Dual Mass Spectrum for each HSL compound identified in the 'Method Blank' (or, Chromatogram of the 'Method Blank' with each peak labelled).
- 3(b). Raw quantification report (i.e., data system print-outs) or integration reports.

[To determine the possibilities of instrument contamination (i.e., carry-over from previous analyses) 'Method Blank' should be analyzed frequently in between sample analyses as specified in the protocol. Lab will provide all documents of replicate 'Method Blank' analyses as described in Sections 3(a) and 3(b) above.]

### 4. Identification and Quantification of the Analyte(s) in the Sample(s) Analyzed

#### 4.1 GC/MS Analysis

If the sample(s) was/were analyzed by GC/MS, the lab will submit the following:

- 4.1a. Information regarding dilution/concentration factor in the extraction of the sample prior to the analysis.
- 4.1b. Unenhanced and enhanced mass spectrum of each HSL compound identified in the sample.
- 4.1c. Quantification report (i.e., data system print-outs) for the sample analyzed.
- 4.1d. Laboratory generated standard spectra for all HSL compounds identified in the environmental samples.
- 4.1e. Identification of the instrument used for the analysis.

#### 4.2 GC Analysis

If the sample(s) was/were analyzed by GC method, the lab will submit the following:

- 4.2a. Information regarding dilution/concentration factor in the extraction of the sample prior to analysis.
- 4.2b. Sample chromatogram with all peaks identified.
- 4.2c. Raw data (i.e., Integration reports) showing retention time and peak area/peak height counts for each identified HSL compound peak.
- 4.2d. Identification of the instrument used for the analysis.

## 5. Precision in Analysis

On each day of analysis, a definite percent of the total environmental samples (to be analyzed on that day) will be analyzed in duplicate.

To establish the precision in the reported results the lab will submit the following documents:

- 5(a). Results and raw data for all duplicate analyses. QC raw data for all duplicate analysis will be the same as described in Section 4 in this report.

## 6. Accuracy of the Results Reported

On each day of analysis at least one 'Method Blank' and a definite % of the environmental samples analyzed on that day will be spiked with a known amount of a 'Spiking Standard Solution', and all spiked samples will be analyzed by using the same analytical methodology and instrument(s) as used in the analyses of environmental samples on that day. Data for review will include the following:

- 6(a). Results and QC raw data for spiked samples analyses as described in Section 4 of this report.

Finally, after reviewing all QC/QA documents as described above the reviewer will apply his professional judgment and experience to evaluate the validity of the reported results.